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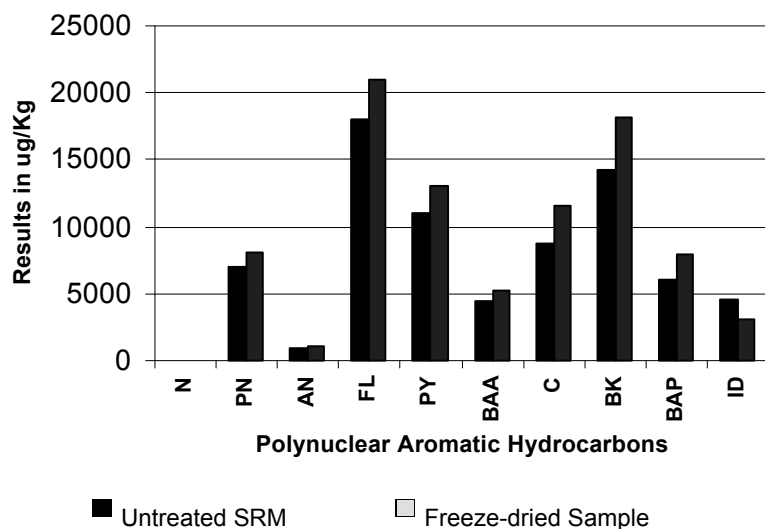
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## SUMMARY

Freeze-drying of sediment samples has been shown to be an effective tool for increasing the solids content of the sediments without adversely affecting the non-volatile chemicals of concern within the samples. This technique can be applied to a wide range of matrices, for example plant tissue, where low solids content has the effect of raising the reporting limits for the chemicals of concern over risk-based criteria. For each batch of samples

undergoing freeze-drying, a reconstituted SRM, if available, should be processed at the same time to serve as a batch quality control sample for the freeze-drying procedure. If an SRM is not available, a matrix spiked sample, may be used for quality control purposes. The freeze-drying procedure should not be used when evaluating "volatile" analytes such as Acid Volatile Sulfides and Simultaneously Extracted Metals (AVS/SEM). In addition, caution should be used if metals speciation (e.g., Arsenic III vs. Arsenic V) is of interest since the freeze-drying process may cause a conversion from one species to another.



**Figure 4.** Results of Native Sediment Sample vs. Freeze-Dried Sediment Sample

## COMPARATIVE ANALYSIS OF SILCOSTEEL COATED SAMPLE PATHWAY AND ELECTROFORM NICKEL SAMPLE PATHWAY IN THE TEKMAR 3100 SAMPLE CONCENTRATOR

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## INTRODUCTION

The Tekmar 3100 Sample Concentrator represents an extension of the 3000 Purge and Trap product line. Silcosteel tubing and Silcosteel-treated sample fittings are used throughout the sample pathway. Silcosteel treatment involves a process that shields the active surfaces of metal. A thin, silicon-based coating is applied to the metal surface, which keeps analytes from adsorbing onto active sites on the metal. As a result, there is enhanced inertness, corrosion resistance, and reduced adsorption. The 3100 Sample Concentrator also features improved temperature uniformity of the heated sample pathway, which reduces carryover, condensation and adsorption. Prior to the introduction of Silcosteel, the sample pathway consisted of electroform nickel and fittings constructed with electroless nickel plating. Difficulties can occur in the analysis of thermally labile analytes, high boiling compounds, flavor volatile organics, and some environmental compounds such as 1,1,2,2-tetrachloroethane. These compounds are targeted for evaluation because many of them are prone to breaking down and adsorbing onto active metal surfaces. In this paper, the results of a comparison study between samples evaluated using a Silcosteel sample pathway and an electroform nickel sample pathway will be presented and discussed.

## EXPERIMENTAL

### Instrumental Parameters

Table 1 describes the Purge and Trap Conditions used for the analyses

Table 2 describes the conditions used for the analysis with a Varian 3800 Gas Chromatograph/Saturn 2000 Mass Spectrometer

**Table 1.** Purge and Trap Conditions

Line Temperature	150°C
Valve Temperature	150°C
Mount Temperature	100°C
Sample Temperature	20°C
MCS Line Temperature	50°C
Purge Time	11 min.
Dry Purge Time	8 min.
Trap Type	VOCARB 3000
Desorb Preheat	245°C
Desorb Time	4 min.
Desorb Temperature	250°C
Bake Time	12 min.
Bake Temperature	270°C

**Table 2.** Varian 3800 GC/Saturn 2000 Ion Trap MS Conditions

Injector	110°C, Split 30:1
Column	J&W Scientific, DB-VRX, 60m x 0.25mm x 1.4µm
Program	45°C (hold 10 min.). Increase to 190°C at 12°C per minute. Hold at 190°C for two minutes. Increase to 225°C at 6°C per minute and hold at 225°C for one minute.
Carrier	Helium at 1.2 mL/min. Constant flow throughout the runs.
Trap Temperature	150°C
Transfer Line Temperature	250°C
Electron Multiplier	1500 volts
Mass Range	35-260

## RESULTS

### Method 502.2 Compound List

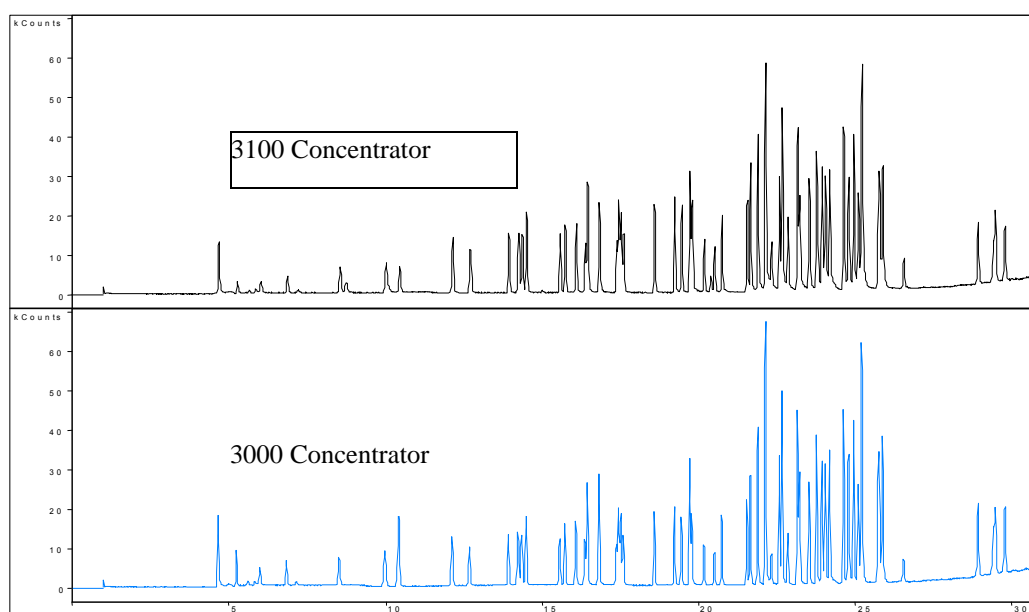
Figure 1 shows the chromatograms obtained for the 502.2 compound mix run at 10 ppb on the Tekmar 3100 and 3000 sample concentrators. Table 3 lists the results obtained for the compound list on the two instruments.

Overall, the 3100 Sample Concentrator yielded higher responses for the analytes in the 502.2 compound list. Naphthalene coelutes with hexachlorobutadiene, but the overall area for the two high boiling compounds is higher when the 3100 Sample Concentrator is used as compared with the 3000 Sample Concentrator.

1,1,2,2-Tetrachloroethane is a component in the 502.2 compound list that has proven difficult in Purge and Trap analyses. Overall the Brominated compounds had a much higher response on the 3100 Sample Concentrator. When the 3100 Sample Concentrator is used, the average response factor shows an increase of about 30%.

### Method 524.2 Rev.4 Polar Compounds

Figure 2 shows the comparison of the polar compounds added in Revision 4 to Method 524.2.



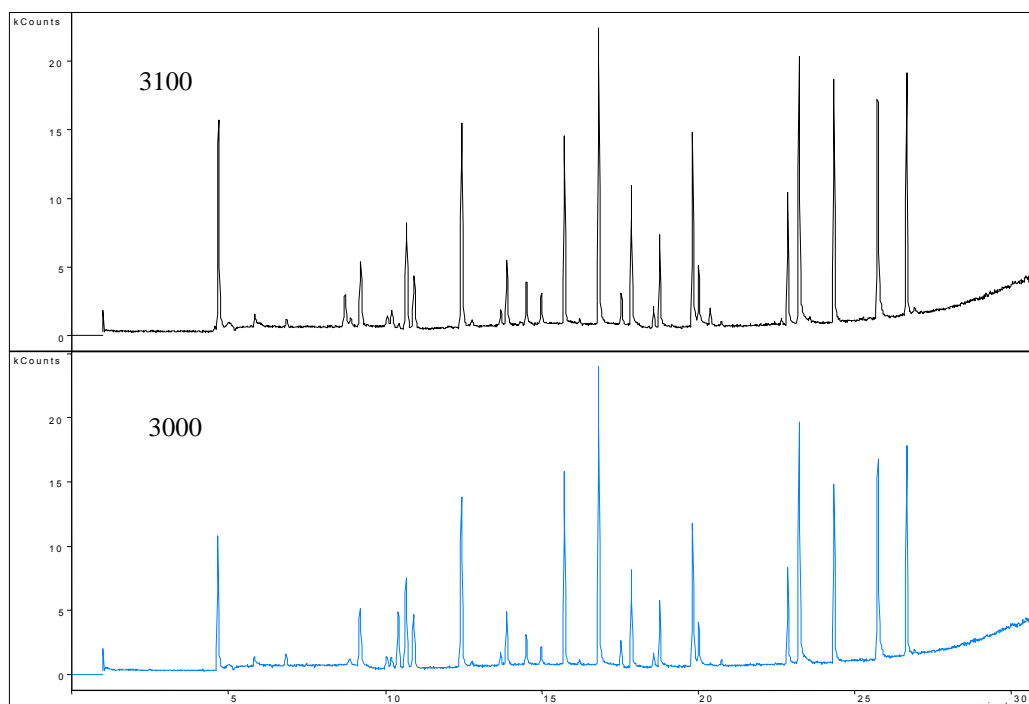
**Figure 1.** Chromatograms for 502.2 Compound List on 3100 and 3000 Sample Concentrators

**Table 3.** 502.2 Compound List Average Response Factor (R.F.) Comparison

	3000	3100	3100
	Avg. R.F	Avg. R.F	% Change
Dichlorodifluoromethane	0.231	0.221	-4.10%
Vinyl Chloride	0.172	0.181	5.20%
Bromomethane	0.243	0.227	-6.60%
Trichlorofluoromethane	0.329	0.355	7.90%
1,1-Dichloroethene	0.472	0.556	17.80%
Methylene Chloride	0.743	0.631	-15.00%
<i>trans</i> -1,2-Dichloroethene	0.573	0.743	29.70%
1,1-Dichloroethane	0.459	0.594	29.40%
<i>cis</i> -1,2-Dichloroethene	0.557	0.705	26.60%
Bromochloromethane	0.553	0.742	34.20%
Chloroform	0.549	0.713	29.90%
2,2-Dichloropropane	0.781	0.986	26.20%
1,2-Dichloroethane	0.481	0.65	35.10%
1,1,1-Trichloroethane	0.649	0.793	22.20%
1,1-Dichloropropene	0.608	0.765	25.80%
Carbon Tetrachloride	0.402	0.494	22.90%
Benzene	1.031	1.377	33.50%
Dibromomethane	0.376	0.474	26.10%

	3000	3100	3100
	Avg. R.F	Avg. R.F	% Change
<b>1,2-Dichloropropane</b>	<b>0.698</b>	<b>1.083</b>	<b>55.10%</b>
Trichloroethane	0.706	0.781	10.60%
<b>Bromodichloromethane</b>	<b>0.481</b>	<b>0.709</b>	<b>47.40%</b>
<i>cis</i> -1,3-Dichloropropene	0.67	0.912	36.10%
<i>trans</i> -1,3-Dichloropropene	0.679	0.949	39.80%
<b>1,1,2-Trichloroethane</b>	<b>0.592</b>	<b>0.873</b>	<b>47.50%</b>
Toluene	1.121	1.23	9.70%
<b>1,3-Dichloropropane</b>	<b>0.6</b>	<b>0.92</b>	<b>53.30%</b>
Dibromochloromethane	0.37	0.523	41.30%
<b>1,2-Dibromoethane</b>	<b>0.305</b>	<b>0.464</b>	<b>52.10%</b>
Tetrachloroethene	0.612	0.757	23.70%
1,1,2,2-Tetrachloroethane	0.71	0.923	30.00%
Chlorobenzene	1.071	1.342	25.30%
Ethylbenzene	1.357	1.69	24.50%
<i>m</i> -Xylene & <i>p</i> -Xylene	2.533	2.891	14.10%
<b>Bromoform</b>	<b>0.301</b>	<b>0.702</b>	<b>133.20%</b>
Styrene	1.074	1.183	10.10%
<i>o</i> -Xylene	1.744	2.263	29.70%
<b>1,2,3-Trichloropropane</b>	<b>0.482</b>	<b>0.923</b>	<b>92.10%</b>
Isopropyl Benzene	1.504	1.633	8.60%
Bromofluorobenzene	1.04	1.213	16.60%
Bromobenzene	1.006	1.343	33.50%
<i>n</i> -Propylbenzene	1.375	1.542	12.10%
2-Chlorotoluene	1.192	1.408	18.10%
4-Chlorotoluene	1.183	1.336	12.90%
1,3,5-Trimethylbenzene	1.4	1.625	16.10%
<i>tert</i> -Butyl Benzene	1.615	1.819	12.60%
1,2,4-Trimethylbenzene	1.324	1.325	0.70%
<i>sec</i> -Butyl Benzene	1.513	1.725	14.00%
1,3-Dichlorobenzene	0.984	1.094	11.20%
<i>p</i> -Isopropyl Toluene	2.561	2.885	12.60%
1,4-Dichlorobenzene	1.788	1.788	0%
Butyl Benzene	1.469	1.609	9.40%

	3000	3100	3100
	Avg. R.F	Avg. R.F	% Change
<b>1,2-Dibromo-3-Chloropropane</b>	<b>0.233</b>	<b>0.368</b>	<b>57.90%</b>
1,2,4-Trichlorobenzene	0.792	0.752	-5.20%
Napthalene/Hexachlorobutadiene	1.19	1.314	10.40%
1,2,3-Trichlorobenzene	0.808	0.729	-9.80%
Sum Average			24.62%



**Figure 2.** 524.2 Revision 4 Polar Compounds

**Table 4.** 524.2 Revision 4 Polar Compounds Average Response Factor (R.F.) Comparison

	3000	3100	3100
	Avg. R.F.	Avg. R.F.	% Change
Acetone	0.039	0.045	15.40%
Diethyl Ether	0.274	0.286	4.40%
Iodomethane	0.067	0.063	-6.30%
Acrylonitrile	0.064	0.088	37.50%
Allyl Chloride	0.413	0.473	14.50%
Carbon Disulfide	0.24	0.24	0%
<i>t</i> -Butyl Methyl Ether	0.72	0.866	20.30%
<b>Propionitrile</b>	<b>0.018</b>	<b>0.036</b>	<b>100%</b>
2-Butanone	0.063	0.05	-20.60%

	3000	3100	3100
	Avg. R.F.	Avg. R.F.	% Change
Methacrylonitrile	0.226	0.226	0%
Methyl Acrylate	0.112	0.144	28.60%
<b>Tetrahydrofuran</b>	<b>0.068</b>	<b>0.126</b>	<b>85.30%</b>
1-Chlorobutane	0.626	0.631	0.80%
Chloroacetonitrile	NR	0.02	N/A
2-Nitropropane	0.085	0.11	29.40%
<b>Methyl Methacrylate</b>	<b>0.296</b>	<b>0.434</b>	<b>46.60%</b>
1,1-Dichloropropanone	0.044	0.062	40.90%
4-Methyl-2-Pentanone	0.23	0.3	30.40%
Ethyl Methacrylate	0.442	0.612	38.50%
2-Hexanone	0.149	0.205	37.60%
<i>trans</i> -1,4-Dichloro-2-butene	0.339	0.411	21.20%
Bromofluorobenzene	0.869	0.899	3.40%
Pentachloroethane	0.659	0.786	19.30%
1,4-Dichlorobenzene	0.804	0.868	8.00%
Hexachloroethane	0.759	0.868	14.40%
Nitrobenzene	NR	0.014	N/A
Sum Avg. Change			23.73%

The 524.2 Rev. 4 polar compounds were also run on a 3000 modified with aged nickel tubing. The chromatogram obtained is shown in Figure 3.

In comparison with the chromatograms and data shown in Figure 2 and Table 4.

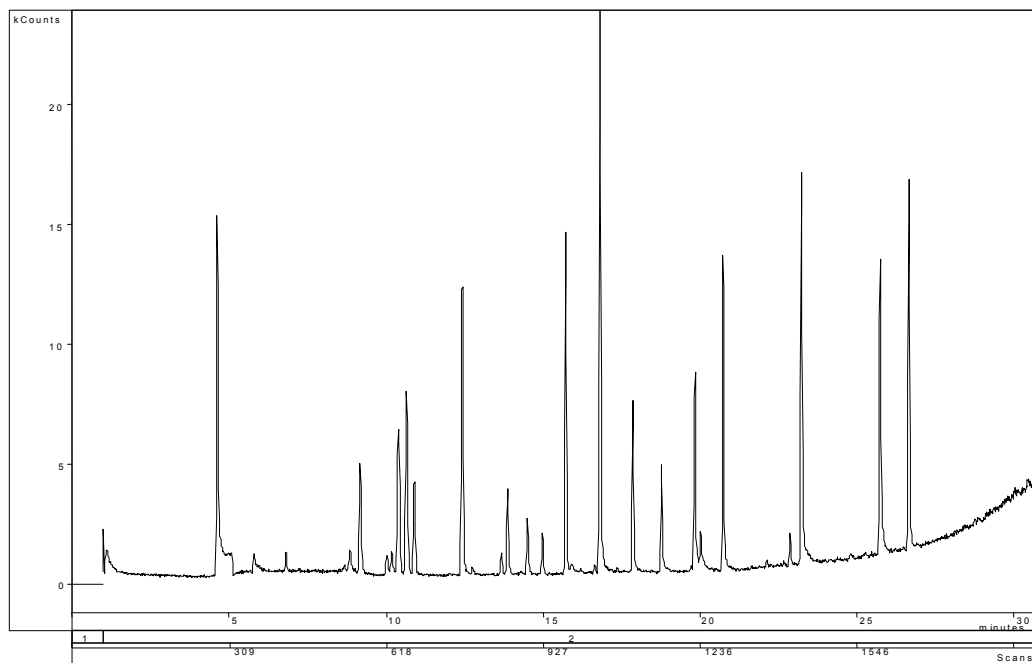
The average response factors obtained with the old nickel tubing are lower than those obtained for the 3000 with newer tubing. For example, diethyl ether has an average response factor of 0.274 with newer nickel. The response factor drops to 0.258 with aged nickel. Similarly, iodomethane shows a drop in response factor from 0.067 to 0.054 with the older tubing. The heavier compounds show an even greater effect with the older tubing, e.g., *trans*-1,4- dichloro-2-butene shows a drop in response factor from 0.339 to 0.056.

Methyl acrylate, 2-nitropropane, 1,1-dichloropropanone, and pentachloroethane completely disappear on the old tubing.

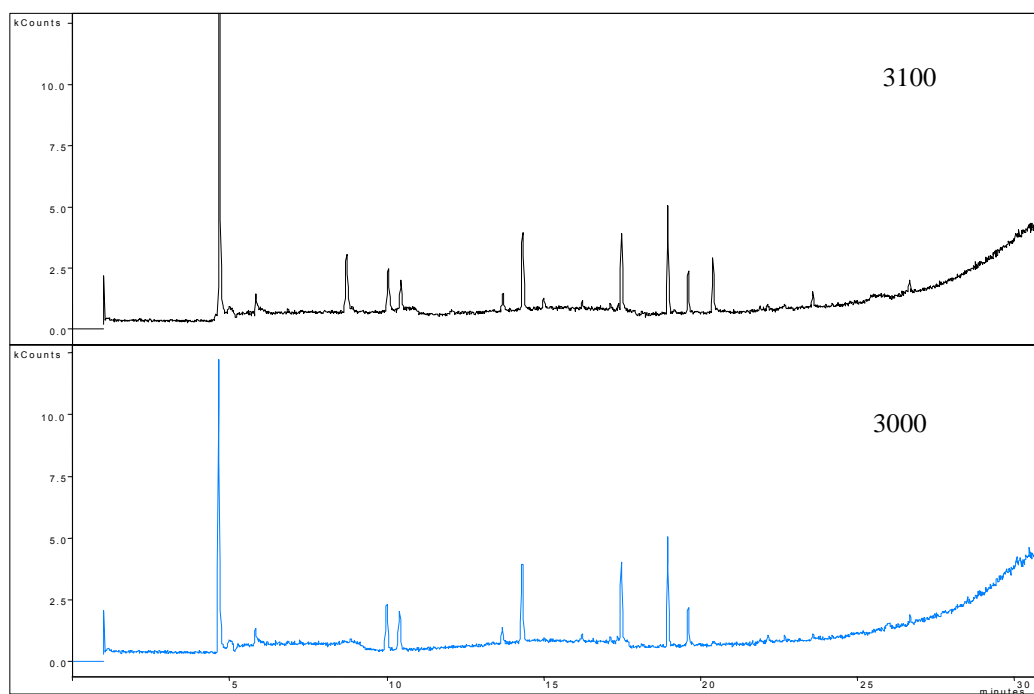
#### Analysis of Sulfur Compounds

A mixture of sulfur compounds at a level of 2 ppb was analyzed on both the 3000 and 3100 sample concentrators. Figure 4 shows the chromatograms obtained.

Table 5 lists the raw area comparisons among the sulfur compounds evaluated on the two instruments.



**Figure 3.** 524.2 Revision 4 Polar Compounds Analyzed on a 3000 Sample Concentrator with Aged Nickel Tubing



**Figure 4.** 2 ppb Sulfur Mix Chromatograms

## CONCLUSIONS

The Tekmar 3000 Sample Concentrator with new nickel tubing performs well in all of the evaluations performed in this experiment. However, when the 3000 is compared with the Tekmar 3100 Sample Concentrator with a complete Silcosteel sample pathway and improved temperature uniformity, the 3100 outperforms the 3000. Average response factors increase by about 30% for a large number of the analytes in the 502.2 and 524.2 Rev. 4 compound lists.



**Table 5.** 2 ppb Sulfur Mix Area Comparisons

	3000 Area	3100 Area	3100 %Change
Dimethyl Sulfide	10075	11161	10.80%
Ethyl Methyl Sulfide	11436	16609	45.20%
Ethyl Sulfide	10777	13738	27.50%
Dimethyl Disulfide	14271	14354	0.60%
Ethyl Thioacetate	5172	5706	10.30%
Sum Avg. Change			18.88%

## FIELD APPLICATION OF ASTM METHOD D5831 AT FUEL CONTAMINATED SITES

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### ABSTRACT

Volatile petroleum products in soil, such as gasoline, can be screened in the field by simple headspace photoionization or flame ionization organic vapor analyzers (OVAs). However, mid-range distillates, such as diesel fuel, and heavier products are not effectively measured by these techniques. In addition, as diesel fuel in soil is subjected to microbial degradation and other weathering processes, the resulting material is less volatile than the original fuel. ASTM Method D 5831, Standard Test Method for Screening Fuels in Soils, provides a procedure for screening soils for diesel-range and heavier petroleum products. This screening method keys in on aromatic components, which are generally accepted as being the more toxic components of petroleum hydrocarbon products with regard to both human health and ecological perspectives. The procedure involves mixing five grams of soil with five grams of calcium oxide, which dries the soil and binds humic materials to minimize interferences. A 50-milliliter aliquot of isopropyl alcohol is added, and the mixture is stirred for three minutes. The extract is filtered, and the absorbance is read at 254 nanometers.

This paper discusses field applications of ASTM Method D 5831 at a diesel fuel spill site and a former aboveground storage tank farm. Comparison of results from split samples that were screened in the field using the ASTM method and analyzed in the laboratory show that the ASTM procedure provided an economic yet effective means to determine the degree of soil contamination at these sites.

### INTRODUCTION

Measurement of fuels and other petroleum products in soils is an important environmental activity. Field screening methods for this purpose are especially useful for defining boundaries of contamination and providing information on where samples should be taken for more expensive and time consuming laboratory analyses. Such methods can also result in cost savings for site excavations by minimizing their duration and the incidence and costs of unnecessary removal of uncontaminated soils.

Volatile petroleum products in soil, such as gasoline, can be screened in the field by simple headspace photoionization or flame ionization organic vapor analyzers (OVAs). However, mid-range distillates, such as diesel

fuel, and heavier products are not effectively measured by these techniques and require a screening procedure that is sensitive to diesel-range and heavier petroleum products. ASTM Method D 5831, Standard Test Method for Screening Fuels in Soils, provides such a procedure (ASTM 1999). This screening method has been tested in the laboratory and shown to recover mid-range and heavier petroleum products from soil (Schabron et al. 1995). Fuels containing aromatic compounds, such as diesel fuel, as well as other aromatic-containing hydrocarbon materials, such as motor oil, crude oil, and coal oil, can be determined using the procedure. This screening method keys in on aromatic components in the contaminant, which are generally accepted as being the more toxic components of petroleum hydrocarbon products with regard to both human health and ecological perspectives. As diesel fuel in soil is subjected to bacterial degradation and other weathering processes, the remaining fuel is more aromatic than the starting material and less volatile (Douglas et al. 1992), making the procedure particularly suited for determining this type of weathered fuel.

The ASTM screening method uses low-toxicity chemicals and can be used to screen organic- rich soils. It is fast, taking about 10 minutes per sample; easy; and inexpensive to perform, costing about ten dollars per test. The procedure calls for mixing a five-gram soil sample with approximately five grams of calcium oxide, which prevents interferences from moisture and humic materials in the soil (Schabron et al. 1995). The mixture is extracted with 50 milliliters of isopropyl alcohol (IPA) for three minutes. The resulting extract is filtered, and the ultraviolet (UV) absorbance of the extract is measured at 254 nm. If the contaminant fuel is available for calibration, the approximate concentration of fuel in the soil can be calculated; if the fuel type is known, but a sample of the contaminant fuel is not available for calibration, an estimated concentration of fuel contamination in the soil can be calculated using an average response factor, which is given in the method; and if the nature of the contaminant fuel is not known, the UV absorbance value is used to indicate the presence or absence of fuel contamination.

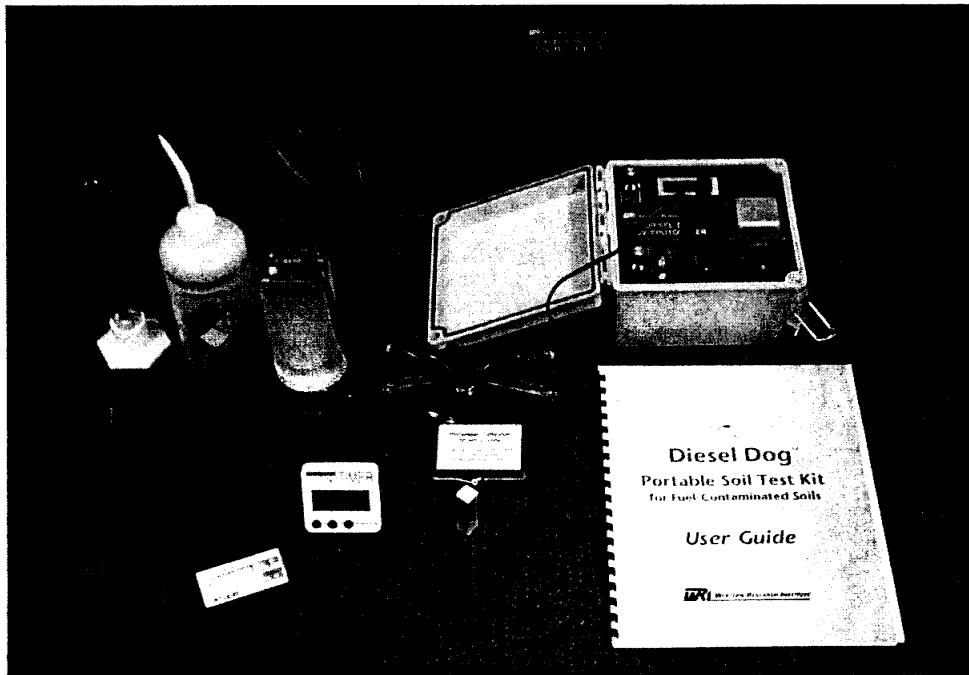
This paper discusses two field applications of ASTM Method D 5831. The ASTM method was used to screen soil samples from a site where diesel fuel was released over a period of approximately 80 years during railroad maintenance activities. The objective was to use the ASTM method to generate field results to guide placement of boreholes and determine the extent of contamination at the site. In a second field application of the ASTM method, soil samples collected at a former aboveground storage tank farm were screened for their concentrations of total petroleum hydrocarbons (TPH). The objective of this activity was to determine the degree of surface soil contamination in the area beneath the tanks in an economic and accurate manner. In both applications, soil samples were screened in the field using the screening procedure and a portable soil test kit. In addition, splits of soil samples were also analyzed in the laboratory, and field and laboratory data were compared.

## EXPERIMENTAL

### Evaluation of a Diesel Fuel-Contaminated Railroad Site

The ASTM method was used at a diesel-contaminated railroad site to help select placement of boreholes and to provide field screening data on the extent of contamination. During three days of sampling, 64 borehole samples were collected and analyzed by the ASTM screening procedure using a portable soil test kit. The components of the portable soil test kit that was used to perform the ASTM method in the field are shown in Figure 1. As described in the method, five-gram soil samples were blended with five grams of calcium oxide in 250-milliliter sample jars. This procedure minimizes interferences from moisture and humic materials that may be present in the soils. The samples were then extracted by adding 50 milliliters of isopropyl alcohol and stirring with a battery operated, magnetically coupled portable soil extractor for three minutes. The resulting extracts were filtered through a 0.45-micron Teflon™ membrane and analyzed using a portable UV spectrophotometer set at 254 nm. Measurements were made relative to standard solutions prepared using fugitive product collected at the site.

Splits of five of the samples that were analyzed using ASTM Method D 5831 were analyzed for extractable petroleum hydrocarbons (EPH) in the laboratory using the Massachusetts Department of Environmental Protection (MA DEP) gas chromatography/flame ionization detection (GC/FID) method (MA DEP 1995). This method involves methylene chloride extraction of the soil in a Soxhlet apparatus. The extract is dried with sodium sulfate, and the solvent is evaporated and solvent exchanged into hexane in a Kuderna-Danish concentrator. The extract is separated into aliphatic and aromatic fractions using a Sep Pak™ cartridge (Waters, Milford, MA) and eluting with hexane and methylene chloride, respectively. The extracts are analyzed using gas chromatography with flame ionization detection. The aliphatic fraction chromatogram is integrated within the C<sub>9</sub> through C<sub>36</sub> aliphatic hydrocarbon range, and the aromatic fraction chromatogram is integrated within the C<sub>10</sub> through C<sub>22</sub> aromatic hydrocarbon range. Total extractable petroleum hydrocarbons is operationally defined as the sum of the C<sub>9</sub> through C<sub>36</sub> aliphatic hydrocarbons and the C<sub>10</sub> through C<sub>22</sub> aromatic hydrocarbons.



**Figure 1.** Components of the Portable Soil Test Kit Used to Perform ASTM Method D 5831

#### Evaluation of a Former Aboveground Storage Tank Farm

The ASTM method was used to screen 11 soil samples collected at a former aboveground storage tank farm for TPH. The soil samples were collected at depths of 15 to 25 cm from areas that were formerly beneath the aboveground storage tanks. A soil auger, which was decontaminated between sampling locations, was used for sample collection. Each sample was stored in a plastic bag, sealed, and labeled for subsequent on-site analysis. The ASTM procedure described above was applied to the samples using a portable soil test kit (Figure 1). Measurements were made relative to standard solutions that were prepared by dissolving the contaminant fuel associated with the site in isopropyl alcohol.

Splits of samples from three of the sampling locations were analyzed in the laboratory: two by gas chromatography analysis using EPA Method 8100 for TPH (U.S. EPA 1996a), and the third by gas chromatography/mass spectrometry analysis using EPA Method 8260 (U.S. EPA 1996b) with methanol preservation for volatile organic compounds (VOCs).

#### **DISCUSSION OF RESULTS**

##### Evaluation of a Diesel Fuel-Contaminated Railroad Site

The petroleum product concentrations in the five samples from the diesel-contaminated railroad site that were analyzed by both ASTM Method D 5831 and the MA DEP EPH method are listed in Table 1.

**Table 1.** ASTM Method D 5831 and MA DEP EPH Method Results for Five Diesel-Contaminated Soils from a Railroad Site, mg/Kg Dry Soil Basis

<u>Sample</u>	<u>ASTM Method D 5831</u>	<u>Aliphatics</u>	<u>MA DEP EPH Method</u> <u>Aromatics</u>	<u>Total EPH</u>
Sample 1	23,000	5,300	2,000	7,400
Sample 2	7,600	870	470	1,300
Sample 3	68,000	44,000	12,000	56,000
Sample 4	13,000	3,400	1,300	4,700
Sample 5	16,000	3,100	1,100	4,300

As shown in Table 1, for all of the samples, the UV absorption results from the ASTM method are higher than the sum of the aliphatic and aromatic hydrocarbons (Total EPH) determined by gas chromatography using the MA DEP method. This result is not surprising since the contamination at the site occurred over a period of 80 years, resulting in extensive weathering and bacterial degradation of the contaminant fuel. It can be assumed that over the years, the aliphatic components were degraded by bacterial action, leaving the aromatic components, which are tightly adsorbed to the soil matrix (Englert et al. 1993). These components can have aromatic structures  $>C_{22}$ , which would not be reported by the gas chromatography method. In addition, isopropyl alcohol is a more powerful solvent for displacing adsorbed polar species than methylene chloride, resulting in a greater extraction efficiency.

Least squares regression analysis using the data listed in Table 1 was performed to determine the numerical relationship between  $C_9$  through  $C_{36}$  aliphatic hydrocarbons versus ASTM Method D 5831,  $C_{10}$  through  $C_{22}$  aromatic hydrocarbons versus ASTM Method D 5831, and total extractable petroleum hydrocarbons versus ASTM Method D 5831. Each of the three regressions was found to be linear using a test for a higher order polynomial relationship at 95 percent confidence. The least squares solution to each linear regression is given by the equations listed below.

- (1) Aliphatics (MA DEP) =  $0.57 \times \text{ASTM}$   $r = 0.96$
- (2) Aromatics (MA DEP) =  $0.16 \times \text{ASTM}$   $r = 0.98$
- (3) Total EPH (MA DEP) =  $0.73 \times \text{ASTM}$   $r = 0.96$

The data indicate that ASTM Method D 5831 is a statistically significant estimator of  $C_9$  through  $C_{36}$  aliphatic hydrocarbons,  $C_{10}$  through  $C_{22}$  aromatic hydrocarbons, and total extractable petroleum hydrocarbons. The slope of equation 3 suggests that the ASTM screening method slightly overestimated the concentration of EPH as measured by the MA DEP method. This may be due to a tendency toward low bias from the MA DEP EPH method because of non-detection of  $>C_{22}$  aromatic components by the GC method or because of loss of some material during the fractionation procedure.

#### Evaluation of a Former Aboveground Storage Tank Farm

The concentrations of TPH determined by screening the samples collected from the soil at the former aboveground storage tank farm using the ASTM procedure are listed in Table 2, along with the results of the laboratory analysis for TPH using EPA Method 8100. For both samples analyzed by EPA Method 8100, the TPH concentrations determined by the ASTM method are about 30 percent less than the values determined using the laboratory method. The values reported from the EPA 8100 analysis are subject to interpretation and are based on measurements made relative to standard solutions prepared using a generic fuel type. In the ASTM method, the absorbance reading of the sample extract is read directly from the photometer. Site-specific standard solutions were used for the ASTM screening method. As a result, the values determined using the ASTM method are based on a milligram of site-specific contaminant per kilogram of soil, instead of being based on a milligram of generic fuel per kilogram of soil. As is most often the case, a sample of the site-specific fuel was not submitted to the laboratory for preparation of laboratory standards. As a result, the TPH concentrations determined using the ASTM method may actually be more accurate than the values determined using EPA Method 8100.

A split of sample 4 was analyzed by EPA Method 8260 for VOCs. The sample was preserved in methanol to prevent loss of VOCs during transport to the lab. The concentrations of benzene, ethylbenzene, and the xylenes (BTEX) in the sample totaled 8.2 mg/Kg, and the other volatiles totaled 7.4 mg/Kg. Comparing these values to the TPH concentrations generated by the ASTM method shows that the contamination in the soil is primarily in the form of heavier fuel products. As a result, screening techniques designed only for detecting volatile components would not have been suitable for screening this site and providing accurate information on the levels of contamination.

#### **SUMMARY**

Field application of ASTM Method D 5831 using a portable soil test kit at a diesel-contaminated railroad site was successful in providing field results for use in guiding placement of boreholes and determining the extent of contamination at the site. Screening samples at the site using the ASTM method and analyzing split samples by the MA DEP laboratory method showed a linear correlation between the results generated by the ASTM screening procedure and the MA DEP laboratory procedure. The ASTM method was shown to give slightly higher results than the state approved laboratory-based method, which is a safe bias when basing decisions on field measurements.

**Table 2.** ASTM Method D 5831 and EPA Method 8100 Data Generated by Testing Soil Samples from a Former Tank Farm

Sample	TPH Concentrations by ASTM Method D 5831, mg/Kg	TPH Concentrations by EPA Method 8100, mg/Kg
1	5,410	— <sup>a</sup>
2	5,540	—
3	37,100	52,500
4	43,200	—
5	14,300	—
6	12,700	—
7	22,200	32,700
8	4,080	—
9	1,260	—
10	15,000	—
11	310	—

<sup>a</sup> No laboratory sample available

Field application of the ASTM method using a portable soil test kit at a former aboveground storage tank farm showed the usefulness of the method when contamination is primarily in the form of heavier fuel products. The contamination at this site was shown to contain very low concentrations of volatile components relative to heavier fuel products, and because this method is designed for determining diesel-range and heavier petroleum products, it was used successfully to determine the degree of soil contamination in the areas that were formally beneath the tanks. The ASTM method may have provided more accurate concentration values than the laboratory TPH method. Concentration values determined in the field using the ASTM procedure were based on measurements made relative to the site-specific contaminant, whereas laboratory data were generated based on measurements made relative to a generic fuel type.

In summary, ASTM Method D 5831 provides a rapid, inexpensive, and accurate method for measuring environmental petroleum in the field. The method focuses on aromatic components, which are generally accepted as being the more toxic components of petroleum products with regard to both human health and ecological perspectives. In both field applications described here, the ASTM method was shown to provide an economic and effective means to determine the degree of soil contamination.

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**ON-SITE ANALYSIS OF NITROAROMATIC, NITRAMINE, AND NITRATE  
ESTER EXPLOSIVES IN SOILS AND ON RANGE SCRAP**

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An economical, on-site analytical method is being developed for frequently detected nitroaromatic, nitramine, and nitrate ester explosives (e.g., TNT, TNB, RDX, HMX, NG, 2,4-DNT, 2,6-DNT, PETN, 2Am-DNT, 4Am-DNT, Tetryl, 1,3-DNB) in soil and in samples associated with range scrap characterization. The method uses a portable gas chromatograph (GC) equipped with a thermionic ionization detector (SRI Model 8610C/TID-1). Soil samples of up to 20 g are extracted by shaking with an equal or greater volume of acetone, and extracts are filtered through a Millex SR (0.5- $\mu$ m) filter. Surface swipes or small pieces of range scrap debris are extracted with acetone. In both cases hardware-store-grade acetone can be used to help reduce shipping and analytical costs. Once extracted, a 1- $\mu$ L volume is manually injected into a heated injection port of the GC. Depending on the data quality objectives, the onboard air compressor may be used to supply carrier gas, eliminating the need for an auxiliary gas supply. Separations of the analytes listed above are achieved during analytical runs of less than 7 minutes. Detection limits of less than 0.010 mg/kg for soil and less than 0.01 mg/L in a solvent extract are easily achieved for many of these analytes. Samples can be prepared and analyzed within 30 minutes.

One of the major advantages of this method, compared with other currently available colorimetric and enzyme immunoassay methods, is its ability to individually quantify suites of target analytes that often coexist on range scrap and in soils contaminated with residues of explosives. For example, this field analytical method will be particularly useful at military training ranges, where it is necessary to quantify residual concentrations of RDX in the presence of high concentrations of NG, PETN and HMX, and when the transformation products of TNT need to be identified. The GC used in this on-site method is commercially available for less than \$10K, and the method typically allows for 35 $\pm$ 10 analyses per day.

This presentation will cover the laboratory development and show the strong correlations that have been obtained when comparing results between the GC-TID-1 and accepted methods of analysis (Methods 8330 and 8095) for a variety of field samples. The first investigation was a field exercise, assessing explosive residues on surface soil/vegetation samples obtained over buried land mines. A second site investigation looked at soil samples taken from a chemical depot where the practice of OB/OD has contaminated the near-surface zone. Another investigation characterized the explosives associated with range scrap debris and included a rapid analysis of QA coupons to be used in a low-cost hot gas treatment system. Lastly, results for soil samples analyzed through the EPA's Environmental Technology Verification Program will be summarized.

# **QUALITY ASSURANCE**





## **DETERMINING AND DOCUMENTING DATA QUALITY (ASTM EFFORT)**

L. Williams

No abstract available.

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## **DOD/ACIL EFFORTS IN DATA QUALITY, PART I**

C. Groenjes

No abstract available.

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## **DOD/ACIL EFFORTS IN DATA QUALITY, PART II**

D. Henderer

No abstract available.

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## **THE ELECTRONICALLY ENHANCED ASSESSOR — WEARABLE COMPUTERS FOR AUDITS**

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### **ABSTRACT**

Results from the first phase of a pilot study of wearable computers for audits were reported at the USEPA's "19<sup>th</sup> Annual Conference on Managing Quality for Environmental Systems," Albuquerque, NM, April 4-7, 2000<sup>1</sup>. That paper described the use of wrist-mounted touch-screen panel monitors and method-specific checklists. This paper presents the second phase pilot study results, using additional data input and processing combinations of wearable computer accessories and software applications to make audits quick, easy, and more comprehensive. These included voice recognition software, head-mounted video and audio display, and document imaging and scanning alternatives. Method-specific checklists, and draft NELAC quality system checklists were applied. Experiences on-site are described and an evaluation of the equipment's performance is reported. Recommendations regarding the effective use of wearable computing systems as assessor tools are offered.

### **INTRODUCTION**

Auditors on-site need quick access to large amounts of information—contracts, QAPPs, SOPs, audit checklists, audit plans, previous visit records, PE scores, correspondence—that can't all be carried in hardcopy. Electronic availability, for easy storage, viewing, or printing, is an answer to auditors' needs. Desirable features of a computerized system that provides this access are:

- Several options for input and output
- Lightweight for extended carry
- Ruggedized for harsh environments
- Easy to assemble and operate
- Expandable with off-the-shelf components
- Battery-powered
- Large memory

Mr. Mills, the principal author, was retained by The Environmental Company, Inc. to lead assessments of environmental analytical laboratories and to conduct field audits during site investigations. He teamed with the sponsor's chemist, Peter Chapman, to select appropriate audit questions and checklists. The authors considered different equipment that might meet these requirements:

- Palm Pilot and other personal digital assistants
- Touchpad portable computer
- Digital voice recorder
- Video camera

These alternatives were found to be partial solutions, because each had various limitations:

- Input/output options—one or two choices at most
- Small memories—up to a few hundred MB
- Battery life of only one or two hours in continuous use
- Not ruggedized for use or transport

The best response to an auditor's needs is a wearable computer system. It is small and lightweight, yet powerful and can be either touch- or voice-activated. They are the perfect tool wherever it is necessary to perform multiple activities simultaneously, where work conditions are too fast-paced, cramped, or otherwise inaccessible for traditional computers. Critical information can be viewed during "heads-up, hands-free" operation of wearable PCs while performing tasks, resulting in higher quality and output in a shorter time, improved safety, and reduced staffing needs. Wearable computers are now available to perform a wide range of audit tasks easily, safely and efficiently, such as

- Accessing specifications while inspecting ongoing operations
- Collecting data and completing QA checklists while observing work in progress
- Following a video or computer-based program or procedure while in the field

Wearable PCs can be integrated into existing auditing systems so auditors can capture information that auditors need on-site, in real time. They can complete and issue checklists, forms, and reports while on-site. The wearable computing platform incorporates wireless technologies to connect periodically or continually to an enterprise. E-mail can be sent and retrieved, as well as video feeds, from remote locations. The accompanying table lists the equipment and specifications available for configuring a wearable system and accessories. It's like wearing a light desktop PC, but not as heavy or awkward. With instant access to a database of methods, checklists, and lab history, the auditor has the flexibility to make modifications on-site. The auditor can capture and record a variety of information, and can produce a report before leaving the site.

Xybernaut Corporation is the leading provider of wearable computing hardware, software, and services. The company's patented wearable computer is a full-function Pentium PC that runs Microsoft MS-DOS®, Windows®, and Windows NT®, along with UNIX, Linux, and other operating systems that run on the Intel x86 architecture. The MA IV® model allows users hands-free access to information in the computer's internal storage, in local area networks and on the Internet on an as-needed, where-needed basis, using consistent navigation techniques and screen presentations. With the MA IV®, customers realize immediate savings in maintenance and repair, diagnosis, inspection, inventory control and data collection procedures. Xybernaut is headquartered in Fairfax, Virginia, with offices and subsidiaries in Germany and Japan. (Visit Xybernaut's web site at <http://www.xybernaut.com>.)

Interface options in addition to the XyberPanel™ touch screen wrist unit include: the XyberKey™ wrist-mounted keyboard used for entering text and numeric data in the field; the XyberViewä miniature, color VGA monitor

mounted on a comfortable headset, suspended in front of the eye without obstructing vision (to the user's eye it appears the same as a 15 inch screen positioned 18 inches away); for hands-free operation, a microphone/earphone can be used to enter voice commands (this component also enables phone communication); the XyberCam™ head-mounted video camera can transmit real-time video images to a remote expert, or record to hard disk for later review, or provide on-the-job training. The computer can receive and send information by radio frequency (RF) Local Area Network (for ranges <1500 feet), by cell phone for medium-to-long ranges, and by satellite relay for specialized applications.

Connecting and configuring the Xybernaut Mobile Assistant IV™ is easy. Components and connectors are designed for wearable use; they all lock into place so they won't slip out as you move around. The MA IV™ can also be used as a standard desktop computer. The Full Port Replicator attaches to the MA IV™ CPU, providing ports for attaching peripherals—such as a CRT desktop monitor, an MA IV™ 11" keyboard, a desktop mouse, and MA IV™ floppy diskette and CD-ROM drives. A PCMCIA network card allows connection to the local network. The Port Replicator has standard sound, parallel, serial, and USB ports.

Mr. Paddock and Mr. Foreman at DynCorp entered the questions from method-specific checklists and NELAC draft checklists into an Access database. They generated templates for asking questions/recording answers, and provided training on the setup and use of the Xybernaut equipment and related software. The following are observations of equipment performance and effectiveness during the overall assessment process (planning, preparation, audit and reporting) that will be helpful in making improvements for future applications to field and lab audits.

#### Results from Phase I Assessment

Results from the first phase of a pilot study of wearable computers for audits were reported at the USEPA's "19<sup>th</sup> Annual Conference on Managing Quality for Environmental Systems," Albuquerque, NM, April 4-7, 2000 in a paper entitled "The Augmented Auditor--The Electronically Enhanced Assessor--Wearable Computers for Audits." The results from the use of the XyberPanel™ flat-panel display and keyboard are summarized in this section.

**Battery Life**—Power from the batteries lasts about four hours. Audit teams should travel with a minimum of two fully charged batteries if possible, and recharge them at every opportunity. Depleted batteries were replaced and recharged during the day, and reused as needed. Smaller, lighter, longer-life batteries are desirable.

**Software Applications**—The checklist templates captured all the Yes/No/NA answers, using handwritten/keystroked input. The templates for each checklist were viewed in report form in Microsoft Access, then exported to Microsoft Word as files for edits. The completed checklists were distributed at the audit debriefing. After brief training, audit team members were able to add or edit questions to modify templates as needed for new topics and procedures.

**Portability**—The XyberPanel™ monitor input device was worn on the wrist, and the hard drive was carried in one lab coat pocket while either wearing the battery clipped to the belt, or in another coat pocket. With a keyboard and additional batteries added in other pockets, the numerous cables could be a safety problem unless wrapped/velcroed down. A backpack or chest harness would be helpful for carrying the equipment comfortably and for easiest access. The XyberPanel™ display was surprisingly readable, even with safety glasses on in bright light, and the stylus made it easy to navigate between programs, folders, and files.

**Configuration**—Using the wrist pad requires the auditor to "train" the SmartWriter™ to recognize his/her handwriting. With a limited number of training sessions, handwriting recognition was only about 50% accurate. This is attributed to the hasty manner in which entries were often made to capture spoken responses. Stylus/keyboard input took the focus of attention away from the individual being queried, and the easy flow of question and answer was slowed. Pointing and double-clicks with the touchpad stylus required some practice. The keyboard was more accurate, but required two-hand use, or an empty benchtop to stabilize it. At least an hour is suggested for handwriting recognition training time per user; more is better. Auditors should train on the particular configuration to be used, in order to get comfortable and proficient with the apparatus.

#### Results from Phase II Assessment

Additional lab and field audits provided further opportunities for testing and refining these audit applications. Portable scanners and digital cameras were used to electronically capture examples of documentation. Voice recognition software responded to queries and recorded responses of lab and field personnel to checklist

questions. Observations about the performance and effectiveness of these wearable computer system components are presented below.

**Battery Life/Power Supply**—Steady use of the voice-recognition software drains the Xybernaut batteries faster than other devices, so more frequent battery switching was necessary. The printer/scanner unit had to be plugged in to AC outlets for use, so materials to be scanned/copied were brought to the unit. The Olympus digital camera batteries were good for 100 images, about two hours.

**Software Applications**—The checklist templates captured the checklist answers, using voice entry. The templates for each checklist were viewed in report form in Microsoft Access, then exported to Microsoft Word as files for edits. The completed checklists were distributed at the audit debriefing. About 4-6 hours of “training” of the speech recognition unit for individual auditors are necessary to obtain a high rate of accurate transcription. The unit can recognize navigation commands from within programs when spoken commands are clearly enunciated. Background noise interferences were filtered out effectively. Audit team members were able to add or edit questions to modify templates as needed for new topics and procedures. The printer/scanner software allowed images to be captured without significant loss of detail, and with a minimum of editing.

**Hardware Applications**—The color printer/scanner performed acceptably, at the rated capacities (see table). Scanned images were of suitable quality and detail for reporting. The Olympus digital camera provided much greater resolution and image quality than the headset video camera, and was used for imaging several documents that couldn't be easily scanned. Images were easily stored and edited. Even the lowest resolution produced readable computer images, each less than 1.4 MB. The headset microphone/video display were comfortable during extended wear. The display was clearly visible even in bright light, while the microphone picked up the voice comments and commands without the need for frequent volume adjustments.

**Portability**—The Xybernaut hard drive with built in mouse buttons was carried in one lab coat pocket or attached to the belt harness. Battery power supply was also clipped to the belt, or carried in another coat pocket. The XyberView™ head-mounted display was easily readable, even with safety glasses on in bright light, and the mouse buttons and/or voice commands enabled quick navigation between programs, folders, and files.

**Configuration**— Using a headset with a microphone for speech recognition, and an eyepiece monitor for video display allowed hands-free input for completion of forms and checklists. Cameras captured video and single-frame shots of the lab areas, documents, people, instrument configuration, etc. during different aspects of the audit. Using the microphone and speech recognition software requires the auditor to “train” the computer software to recognize his/her personal speech patterns. With a limited number of training sessions, speech recognition was only about 85% accurate. At least four hours are suggested for speech recognition training time per user; more is better. Auditors should train on the particular configuration to be used, in order to get comfortable and proficient with the apparatus.

**NELAC Draft Checklists**— Audit templates and method-specific checklists were compiled and stored on the hard drive of the wearable computer system. These included draft NELAC quality system checklists that are publicly available. These checklists cover instrumentation categories in general, with some method-specific questions for GC, AA, ICP, and GC/MS and wet chemistry. They have common elements. The quality system checklist is extensive and unwieldy, and could be simplified.

## **SUMMARY**

Assessment teams can benefit from using wearable computers:

1. Fast input and output. The team can collect information by scanning documents, by digital pictures, by voice, handwritten, or keystroked input, and assemble it in template checklists or freeform reports. A complete report can be presented at the debriefing to allow the audited organization to begin responding and taking corrective actions immediately.
2. Access to information. Auditors can package many checklists, SOPs, contracts, QAPPs, FSPs, HASPs, etc. covering a variety of topics (methods, surveys, old reports, problems, etc.) and have instant access to them on the hard drive. New information can be added from diskette or CD-ROM and compared to historical records.
3. More flexibility. With instant access to an electronic database of methods, checklists, and lab history, the on-site audit team can quickly and easily modify an audit plan, checklists, and reports. A wearable

computer system can enhance the abilities of assessors to quickly and accurately collect and report information about an auditee's capabilities and performance.

4. More effective communication. A team may use cell phones, video cameras, or radios to communicate and share information as needed immediately, and can link with off-site contacts. Using the wearable computing system, the team can take pictures of the entire operation's equipment, personnel, and facilities, record observations made during the assessment, and provide audio commentary as well as a video and hardcopy report. "Virtual audit" records will become increasingly important for those who want to know as much as possible about the audited facility, but weren't present.
5. Cost effective. A wearable computer system, with accessories and software applications that are easily operated and simple to maintain, can pay for itself by simplifying or eliminating traditional audit procedures.

Estimated Savings—for an example SDWA State Primacy Audit:

4-man team, 5 days on-site + 50 hours for report preparation = \$7,350 labor, \$3,170 expenses (airfare, rental car, lodging, meals)

Same team using wearable computer equipment could finish in 4 days instead of 5, and furnish a report at debriefing, saving an estimated \$2,320 (22%)

Greater savings may be achieved with practice and an established repository of templates

### **For Phase III—The Future**

Using the wearable computing system, an auditor or team could take pictures and record observations of the entire lab and its operations, edit in the observations made during the assessment for audited processes, show the equipment, personnel, and facilities, and provide commentary. A virtual lab tour of each area, personnel, instrumentation, facilities, etc. could be prepared, with hyperlinks of lab audit questions to visual evidence and answers. It can be made interactive, with point-and-click capability for each lab area, instrument, person, test method, etc.

Click on an instrument and see calibration and maintenance and run log records.

Click on an individual and see a personnel folder, with resume, proficiency test results, PE sample performance, and training records.

Click on an area of the lab, and see sample flow, testing types, backlogs, etc.

Click on a method and see productivity, control charts, example reports, etc.

Customers could get all the benefits of the audit, without the travel time and expense. The Xybernaut® gear and audit checklists can be used in conjunction with IPIX®-type videos to provide a complete CD-ROM package for auditors, and for customers of audits (regulators, potential clients). Followup and updates with individual clients could use video conferencing to examine a particular area or item in more detail, in real time.

### **Acknowledgment**

Thanks to The Environmental Company, Inc. for sponsoring the pilot study of the application of wearable computer equipment as part of the lab audit process. Thanks to Dyncorp for providing the equipment to Mentorprises Corporation and The Environmental Company, Inc. for this pilot study. Dyncorp is a marketing partner with Xybernaut, and Xybernaut equipment is listed on the Government Services Administration schedule. Contact Dave Paddock of Dyncorp at [paddock@dyncorp.com](mailto:paddock@dyncorp.com) for further information, a demonstration, or to configure a system specific for your needs.

COMPONENT	SPECIFICATIONS
CPU MODULE WITH XYBERPORTS™	<p>Dimensions: 7.4 x 2.5 x 4.6in. (18.7 x 6.3 x 11.7 cm)</p> <p>Weight: approx. 1.9 lbs. (900 g)</p> <p>Processor: 200 or 233 MHz Intel Pentium® II MMX</p> <p>Memory: Up to 128 MB SDRAM</p> <p>Storage: Up to 6 GB internal removable HDD</p> <p>Self-contained, environmentally sound design</p> <p>Shock-mounted hard drive</p> <p>Magnesium alloy case</p> <p>Built-in mouse</p> <p>Built-in dual PC card readers (CardBus)</p> <p>Built-in sound card, full-duplex</p> <p>Ports for HMD/FPD, power, USB and replicator</p> <p>Full port replicator with microphone in, headphone out, line-in, serial, parallel, VGA, PS/2 Mouse, PS/2 keyboard and USB ports</p> <p>Miniport replicator (keyboard and FDD)</p> <p>Speech recognition engine included (IBM's Via Voice and Dragon's Naturally Speaking)</p> <p>Choice of operating system included (Windows 95®, Windows 98®, Windows NT® and others)</p>
HEAD-MOUNTED DISPLAY (HMD) XYBERVIEW™	<p>Weight: approx. 1 lb. (470 g)</p> <p>640 x 480 color VGA monocular</p> <p>Left- or right-side wearable</p> <p>Over- or under-viewable</p> <p>Microphone and ear-piece speaker</p> <p>Optional integrated miniature video camera, XyberCam™</p>
FLAT-PANEL DISPLAY (FPD) XYBERPANEL™	<p>Dimensions: 7.3 x 4.6 x 1.2 in (18.4 x 11.7 x 3.2 cm)</p> <p>640 x 480 color VGA</p> <p>Built-in programmable buttons</p> <p>Pen or touch screen</p>
BATTERY	<p>Weight: 1 lb. (454 g)</p> <p>Lithium-ion (no memory effect)</p> <p>Rechargeable hundreds of times</p> <p>Up to 4 hours of battery life</p> <p>Combined AC power adapter/battery charger with protective circuitry</p> <p>Hot swappable—change batteries without shutting down applications</p>
BJC-85 PORTABLE COLOR PRINTER/SCANNER	<p>Prints 720 x 360 dpi black and color documents from 2 ppm (color) – 5 ppm (black) sheetfed letter or legal-size pages per minute. Scans at 360 dpi with IS-12 Color Image Scanner Cartridge. Uses either a USB or a parallel port connection, or a wireless infrared connection. Up to 200 pages per battery charge. 120V AC, 60 Hz power source required.</p>
OLYMPUS D-500L DIGITAL CAMERA	<p>1.3 Megapixel resolution, 3X optical zoom, menu display, flash and red-eye reduction.</p> <p>640 x 480 pixels standard quality, 48 jpg per 4 MB</p> <p>800 x 600 pixels high quality, 20 jpg per 4MB</p> <p>1024 x 760 super high quality, 6 jpg per 4 MB</p> <p>SmartMedia card storage, 4 MB-32 MB</p>

## **WHEN DOES QA REALLY ASSURE DATA QUALITY?**

C. Carter

No abstract available.

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## **PERSPECTIVE ON CHANGES IN QUALITY STANDARDS**

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The development of Quality Standards from the lack of standards in the past, to many standards at the present, to a unified standard in the future will be discussed. In the past there were few Quality Standards for analytical testing. Now there are many different Quality Standards. These developed in response to different stimuli such as the need to provide data of known quality suitable for a decision, the need to prevent testing failures, or laboratory misconduct. Different agencies and programs independently developed standards. Presently the provider of analytical data is faced with responding to different standards with similar but different requirements. The development of Quality Standards continues and the similar but different and conflicting standards can evolve into a unified Quality Standard if the current direction of development continues. The current situation and the history of the development of some current Quality Standards such as the Good Laboratory Practice Standards, the Drinking Water Laboratory Certification program, the Contract Laboratory Program and EPA's Quality System will be discussed. The activities and stimuli that could lead to a unified Quality Standard such as a Performance Based Measurement System, the adoption of consensus standards such as ISO17025, Interagency cooperative efforts, and the National Environmental Laboratory Accreditation Conference will be discussed.

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## **IMPLEMENTING MQOS WITHIN A DQO FRAMEWORK**

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This paper will describe work in progress on a DOE-funded project that is developing a new Measurement Quality Objectives (MQOs) module to an existing sampling design software package called Visual Sampling Plan (VSP). This module is needed because EPA is implementing a performance-based approach to specifying analytical testing requirements called PBMS (Performance Based Measurement System). Under PBMS, the Agency would identify relevant performance characteristics of analytical methods (e.g., bias, precision, selectivity, sensitivity, etc.), and would encourage projects to use the Data Quality Objectives Process to specify quantitative performance criteria for each of these characteristics (called the project MQOs) without prescribing specific procedures, techniques or instrumentation. The challenge comes when projects need to trade-off increased/decreased sample size for decreased/increased analytical accuracy and precision in order to meet the overall goals of the project as identified in the MQOs and DQOs for the project.

Developing MQOs from DQOs is a topic being addressed by many different groups, including the Quality Division at EPA, the Multi-Agency Radiation Laboratory Assessment Program (MARLAP), individual Program Offices within EPA, and others. The module we plan to add to VSP will evolve as these methods evolve and become industry

standards. For the initial version of the software, it will use reference tables of performance characteristics of standard analytical methods, sample sizes based on user-specified decision error limits, and costs try to find an acceptable trade-off of cost/performance to risk. The MQO module will allow users to modify the inputs to determine whether improved overall performance is achievable. The software will be visual, user-friendly, include method reference tables, and show "what if" scenarios in table form.

The MQO module will be part of VSP, a DOE-sponsored software package developed by the Pacific Northwest National Laboratory for identifying the number and location of samples required to meet user-specified decision criteria.

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## **FIELD TEAM AUDITING AS PART OF THE ENVIRONMENTAL DATA QUALITY ASSURANCE PROCESS**

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Environmental data quality improvement continues to focus on analytical laboratory performance. For example, the current excitement regarding Performance Based Measurement System (PBMS) processes focuses on the opportunity to economically improve laboratory performance (the sample analysis team) with little, if any, attention given to improving the performance of the field crew (the sample collection team). The naïve and often incorrect assumption made by many professionals in the environmental data quality assurance field is that the primary opportunity for data error lies within the walls of a laboratory. Experience indicates that an often-ignored factor affecting environmental data quality is the manner in which a sample is acquired and handled in the field. Field auditing is becoming a more often-employed technique for examining the performance of the environmental sampling field team and how their performance may affect data quality.

In the broadest sense, field auditing of an environmental sample collection contractor is the beginning of the environmental data quality assurance process. Even if laboratory analyses meet all predefined data quality objectives and PBMS criteria, if the sample has been improperly collected, decisions based on the analytical results will be erroneous. Poorly collected and preserved environmental samples result in poor quality environmental data. Poor quality environmental data may result in inappropriate decisions regarding remedial action. In addition, improperly collected samples could result in the mischaracterization of environmental risks posed by a site.

Third-party field audits afford responsible parties an opportunity to demonstrate that environmental samples are being properly collected, packaged, and preserved in the field. In addition, a field audit can verify that the sample is truly "representative" of an environmental media, that investigation-derived wastes are properly managed (cost control), and that proper health and safety protocols are being followed (liability control). Field equipment used to collect a sample can be evaluated in terms of decontamination, suitability for the intended purpose, and whether or not the sampling tool is being properly used.

A third party can observe deviations from Sampling Analysis Plans and Quality Assurance Project Plans and propose practical corrective measures to be implemented if necessary. The cost savings realized by improved waste management, efficient field team operations, sampling equipment use, and the verifiable integrity of a sample as it is collected in the field often pays for the field audit. Rather than examine field team performance after sampling, a field auditor can do so while the samples are being collected, applying corrective action as appropriate.

The cost savings and improved data quality that result from properly completed field audits makes the field auditing process both cost-effective and functional.



## UTILIZING FIELD SCREENING TO MEET OBJECTIVES DURING CHARACTERIZATION AND EXCAVATION OF ALLEN HARBOR LANDFILL

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The presence of PCB contamination at the north end of the Allen Harbor Landfill was identified in 1998 during routine sampling activities on the landfill closure project. The site, located in North Kingstown, RI, was formerly used from 1942 to 1972 for the disposal of waste material generated by the Naval Construction Battalion Center (NCBC), Davisville. The waste material consisted of municipal-type waste, construction debris, rubble, preservatives, paint thinners, degreasers, PCBs, asbestos, ash, sewerage sludge, mineral oil and fuel oil. Upon discovering the PCB contamination, Foster Wheeler Environmental Corporation (FWENC) was tasked with delineating the horizontal extent of contamination at the north end of the landfill and developing several alternatives for remediation of the impacted area. Of the various alternatives presented to the Navy by FWENC, complete excavation and offsite disposal of PCB contaminated soil which exceeded the State clean-up standards was selected. The objective of the delineation activities was to determine which areas of the landfill contained PCB-contaminated soil above the State clean-up standards in order to develop cost and schedule proposals to complete excavation and offsite disposal. To accomplish this objective, FWENC used RaPID Assay (RA) immunoassay PCB test kits (SW-846 Method 4020) from Strategic Diagnostics Inc., with a percentage of the samples also being sent to an off site laboratory for confirmatory analysis. Delineation samples were collected from approximately 175, 20-foot by 20-foot grids. The results of the site delineation sampling demonstrated a strong correlation between the field and laboratory analyses, and the regulatory agencies allowed FWENC to continue using immunoassay testing for confirmation sampling into the excavation phase of the project. Results obtained from the site delineation sampling were combined with existing PCB analytical data to create an excavation plan for 1) PCB soils with concentrations greater than or equal to the TSCA limit of 50 PPM; and 2) greater than the State clean-up criteria of 10 PPM, but less than 50 PPM. Excavation activities were then performed in each of the contaminated grids. At the completion of each one-foot excavation interval, a confirmation sample was collected from each of the grid's four sidewalls and bottom to confirm that the State clean-up criteria had been achieved. A total of 550 confirmation samples were analyzed using immunoassay and 65 were analyzed by laboratory during excavation. Lab analysis of all 550 samples would have cost \$66,000. Correlation between immunoassay and lab method 8082 remained excellent throughout the project. Through use of immunoassay test kits, results of the confirmation sampling were received within the same day that sampling was performed, which increased the rate at which FWENC could excavate and dispose of contaminated soil. The increased production rate enabled the project to be completed approximately fifteen days ahead of schedule, and under budget. Project cost savings resulting from reduced analytical, equipment and manpower requirements were in excess of \$100,000.

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## TRACKING TRENDS ON PROFICIENCY TESTING STUDIES – A HELPFUL TOOL FOR ASSESSING AND IMPROVING DATA QUALITY

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### Abstract

This paper provides a procedure for tracking trends on proficiency testing (PT) studies using commonly available software. By evaluating results for multiple PT studies, trends or anomalies can be identified that would not be indicated in an individual study. Corrective or preventive action taken on trends can help to correct any biases in the test procedure and to prevent future analyses from being out of control. This procedure has been successfully used for a two-year period to track trends on multiple tests by multiple laboratories. Combined with other traditional quality assessment measures, the tracking of trends on PT studies aids in the effort to assess and improve data quality. Project managers, laboratory accreditors, laboratory managers and quality assurance

professionals can use this approach, combined with other techniques, as an innovative tool to track data quality.

## **Introduction**

Proficiency testing studies are used to evaluate laboratory performance on specific analytical tests. PT samples are defined in the National Environmental Laboratory Accreditation Conference (NELAC) Standards as samples of unknown composition to the analyst that are used to test whether analytical results can be produced within specified acceptance criteria. If test results for PT samples are within the study acceptance limits, then laboratory performance is considered acceptable and no action is generally taken. If any test result exceeds the acceptance limits, then the need for corrective action is indicated. Significant and repeated exceedances, combined with other quality control (QC) problems, may result in possible qualification or rejection of affected data or the laboratory until acceptable results can be achieved. A significant number of samples as well as laboratory revenue can be affected from the time the system went out of control until the time that the test is in control again.

In addition to using PT data to evaluate individual sample results, PT data from multiple studies can also be used to monitor trends (upward rises, downward falls, positive bias, negative bias and periodicity) and prevent outliers. While all laboratories should be concerned with outliers, this is especially important for laboratories applying for or wishing to maintain accreditation under NELAC and other regulatory or non-regulatory programs that require acceptable performance on recent PT studies (e.g., two out of three). Removal of accreditation status for repeated unacceptable PT results would not be desirable to a laboratory or their data users. By tracking performance on PT studies over time, combined with other QC measures, trends can be identified and preventive action taken if needed to improve overall data quality and prevent the occurrence of future outliers.

## **Background on Control Charts**

A control chart is a graphical representation of the output of a process, showing plotted values of some statistic gathered from that output, a central line, and one or more control limits. A control limit is a line on a control chart that represents the maximum extent of variation in the statistic being plotted that could reasonably be expected to occur. For this paper, control charts are used to plot the results of laboratory PT studies over time. Although control charts should be maintained and interpreted in real time (such as for laboratory control samples), this paper addresses an alternative use of control charts for additional interpretation of PT study results along with other traditional quality control (QC) techniques.

Control charts are used as tools for determining an acceptable level of laboratory performance, achieving the acceptable level defined, and maintaining performance at that level. Control charts can be used to measure quality characteristics, the percent out-of-control data in a data set, and the number of out-of-control data for a given test. Depending upon the scope of performance evaluation, control limits may be based upon the capability of the process itself or defined by the applicable method, project or regulation. The frequency and evaluation of plotted information is specific to the end-user. Real-time charting, outlier tracking and historical trend analysis generally address various aspects of statistical processing and control.

There are two types of control charts: one focuses on determinations of accuracy and the other on precision. For this paper, only accuracy charts are considered and precision charts will not be discussed. A Shewhart control chart or means (X) chart is an accuracy chart that compares plotted values against an established mean in relation to statistically derived limits. The accuracy of an analyst, analysis and/or test can be evaluated by plotting determined/observed measurements of test samples (including spikes, standards, check samples and other fortified samples). An accuracy chart looks at plotted values in relation to the centerline (mean) and warning and control limits. The centerline, however, may not only reference the mean of historical determinations but also may be the actual true value of the substance analyzed or an arbitrary number. Control limits are typically set at the 95% and 99% confidence intervals or the mean  $\pm$  two (1.96) sigma for the warning limits and  $\pm$  three (2.58) sigma for the control limits, respectively.

Once control charting is set up for a given method, instrument or analyst, evaluation of the plots and determination of the appropriate preventive/corrective action for anomalies may commence. Analysts, laboratory supervisors and quality assurance (QA) personnel should evaluate control charts for out-of-control data and anomalies on a regular basis. Attention to the information provided by control charts may be the difference between a potential nonconformance and an out-of-control event. Emphasis should focus on proactive or preventive actions rather than curative or corrective actions.

Statistical anomalies may be described as suspicious or out-of-control occurrences. Anomalies are determined by

the evaluation of plotted values in comparison to the specified warning and control limits. Both single points and points-in-series may fall into the category of being suspicious or out-of-control. These data serve as evidence to outliers, runs, trends and periodicity. Corrective and/or preventive action should be taken on each.

**Outliers.** There are two types of outliers: a single point that falls outside the warning limit and a single point that falls outside the control limit. A point outside the warning limit but within the control limit is suspicious in nature and, as a proactive measure, should be investigated as such. A point outside the control limit is out-of-control and must be evaluated through corrective action. Recurring or multiple outliers indicate that the initial corrective action may not have been sufficient and that additional immediate action is required. Outliers may be caused by instrument failure, inaccurate calibrations, analyst error, contamination, out-of-tolerance standards and reagents, and other problems.

**Trends.** An unbroken series of 4-6 or more points in an upward orientation (rise) or downward orientation (fall) is considered a trend. This pattern is suspicious in nature and, if uncorrected, could lead to a nonconformance or outlier. Trends may be caused by the degradation or concentration of standards/reagents and by changes in instrument sensitivity or performance.

**Bias.** A series of 6-8 or more points that line up on one side of the mean or centerline is considered a bias. This pattern is suspicious in nature, and, if uncorrected, could lead to a nonconformance. A bias may be caused by analyst error, contamination of the substance being analyzed, incorrect preparation or dilution of standards/reagents, and instrument problems.

**Periodicity.** A recurring pattern of change in plots in equal intervals of unspecified length or amplitude is considered periodicity. This pattern is suspicious in nature and, if uncorrected, could lead to a nonconformance. Periodicity may be caused by cyclic activities inherent in the technical procedure, matrix interferences via recurrent sampling events, and other complex occurrences.

**Preventive/Corrective Actions.** In the event of an out-of-control incident (outlier, beyond the control limit), the affected data should be evaluated and appropriate action taken. If the outlier is known in real time, then corrective action may be taken immediately, including re-analysis of the outlying substance in question. If re-analysis confirms the out-of-control event, then the analysis run may be suspended until the cause is determined and corrected. In the event of a potential nonconformance or knowledge of an outlier after the fact, an investigation as to the cause of the suspicious circumstances should be carried out. This may include re-calibrations, instrument servicing, preparation of new standards and reagents, re-training of personnel and other appropriate actions. All statistical anomalies should be documented via a preventive/corrective action report, marked as a nonconformance or potential nonconformance, and submitted to management and the QA department immediately.

### **Control Chart Procedure for Monitoring Trends**

Core Laboratories' petroleum testing laboratories (including Saybolt Inc.) analyze reformulated gasoline (RFG) samples for specific tests required under the Clean Air Act – 40CFR Part 80. Accurate laboratory performance on these tests is important for ensuring that RFG used for fuel in the United States meets the standards of the Clean Air Act and that emissions are controlled. Inaccurate testing could mean that off-spec fuel is used resulting in regulatory violations and increased air pollution. Proficiency testing samples are used to monitor laboratory performance on regulated parameters, including aromatics, benzene, distillation, olefins, oxygen (oxygenates), sulfur, and vapor pressure. U.S. EPA and American Society for Testing Materials (ASTM) methods are used for testing.

A control chart program developed by Environmental Business Strategies of Cambridge, MA (daryl@alum.mit.edu) using Microsoft Access software has been used for tracking laboratory performance on PT studies. ASTM or other interlaboratory exchange programs (round robins) are used as proficiency test samples for a variety of tests and petroleum products. For RFG analyses, the ASTM Interlaboratory Crosscheck Program is used on a monthly basis to test the required parameters. Though not required by EPA regulation, monthly crosscheck testing is an industry practice for petroleum laboratories that test RFG.

Results for each study are entered in the control chart program each month. The study mean, standard deviation and Z score (individual result – study mean / study standard deviation) from the ASTM report, as well as the reproducibility value of the test method are entered into the control charting program. ASTM flags any values with a Z score exceeding  $\pm 2$  and the control chart reflects this criteria for use in determining acceptability. Control

charts and tables that display the entered and calculated data are generated each month. Data are evaluated and action taken as needed for any result(s) with a Z score exceeding  $\pm 2$ . Corrective action is required for any result(s) with a Z score exceeding  $\pm 3$ . In addition, data are evaluated over time for trends, biases and periodicity. If any of these anomalies are identified, preventive action is initiated to improve data quality and prevent future outliers.

Figures 1 and 2 provide examples of the control chart program output, showing tabular and graphical representations of actual GC and GC/MS data that identify an upward trend and negative bias in monthly ASTM RFG round robin data, respectively. Figure 3 provides an example control chart trend analysis report used to document and act upon identified trends.

The control chart program has been used to track data from monthly RFG crosscheck samples tested by multiple testing laboratories for over a two-year period. In addition to identifying the need for corrective action on outliers, this procedure has been used to identify trends and initiate preventive action. For example, the following trends have been identified for various parameters:

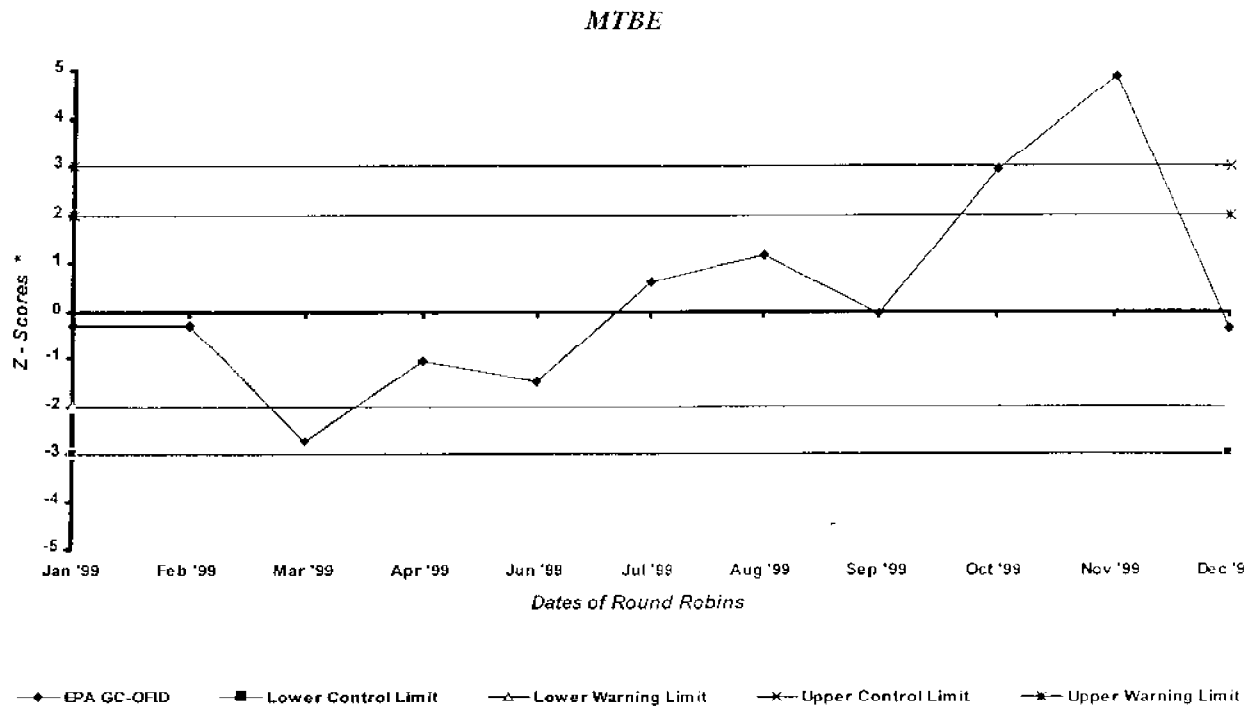
- Successive points in a upward or downward direction indicating a positive or negative trend
- Successive points on one side of the mean indicating a positive or negative bias
- A recurring pattern of change in plots of equal intervals indicating periodicity
- Multiple points in succession outside the control limits (recurring outliers)
- More than one-third points outside the control limits (multiple outliers)
- Identical data for multiple laboratories indicating coincidence or possible replication of data

Identification of these trends has assisted in the initiation of preventive action, the prevention of potential outliers and improved data accuracy.

## Conclusion

The use of a control chart program to track trends identified on monthly crosscheck studies has enabled the implementation of preventive action as well as corrective action to improve data quality on RFG testing required under the Clean Air Act. Commercially available software that can produce control charts can be used to track data. The use of monthly testing helps to ensure that acceptable analytical results can be produced on a regular basis. It is recommended that laboratories needing to maintain acceptable PT performance in order to obtain or retain NELAC or other accreditation consider the use of monthly PT samples, as well as the tracking of outliers and trends.

The tracking of trends on proficiency testing studies is an additional tool that laboratories and evaluators of laboratories can use to measure and help improve data quality. Laboratories can use information on trends, as well as outliers, to improve performance and prevent future outliers. Laboratory accreditors and clients can have increased confidence in laboratories that not only take corrective action on PT study outliers but also take preventive action on identified trends. This approach not only helps to prevent the occurrence of future outliers but also improves overall data quality by continually focusing on the accuracy of the test measurement. Limitations include the time needed to obtain sufficient data to monitor trends, the number of sample tests needed to prepare control charts, employee attitudes towards control charts and their usefulness, and laboratory acceptance of the need for corrective as well as preventive action.

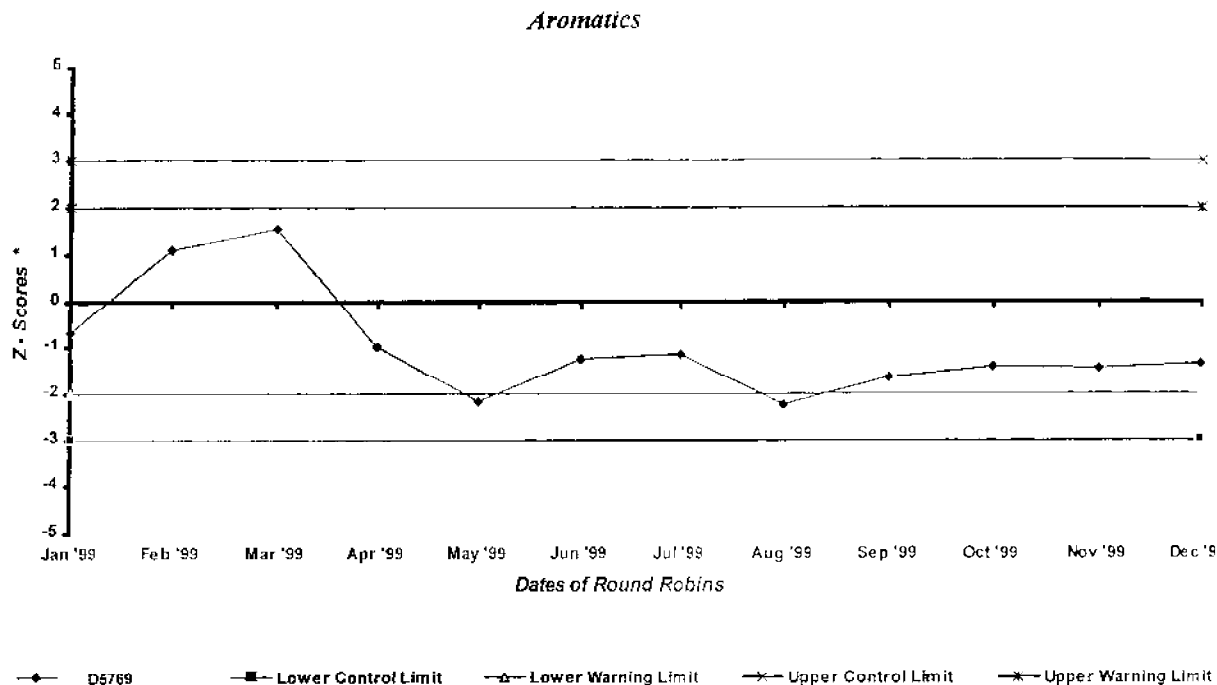


\* Note: Z-Score = (Individual Lab Result - Robust Mean) / Robust Standard Deviation). Preferred Z-Score values are greater than -2 and less than +2.

Date	Method	Mean	Standard Deviation	Lab Result	ASTM Reproducibility	Z-Score	% Recovery	Difference From Mean
<b>MTBE</b>								
01/1999	EPA GC-OFID	11.229	0.28	11.14	0.776	-0.32	99.2	0.089
02/1999	EPA GC-OFID	7.639	0.234	7.57	0.648	-0.29	99.1	0.069
03/1999	EPA GC-OFID	14.712	0.381	13.66	1.055	<u>-2.71</u>	93.0	1.032
04/1999	EPA GC-OFID	6.257	0.194	6.05	0.537	-1.07	96.7	0.207
06/1999	EPA GC-OFID	13.712	0.31	13.25	0.859	-1.49	96.6	0.462
07/1999	EPA GC-OFID	11.324	0.274	11.49	0.759	0.61	101.5	0.166
08/1999	EPA GC-OFID	9.866	0.211	10.11	0.584	1.16	102.5	0.244
09/1999	EPA GC-OFID	9.831	0.171	9.82	0.474	-0.06	99.9	0.011
10/1999	EPA GC-OFID	11.01	0.233	11.7	0.645	<u>2.96</u>	106.3	<u>0.690</u>
11/1999	EPA GC-OFID	9.754	0.246	10.95	0.681	<u>4.86</u>	112.3	<u>1.196</u>
12/1999	EPA GC-OFID	9.756	0.205	9.68	0.568	-0.37	99.2	0.076

Sponsors: ASTM Interlaboratory Crosscheck Program

**Figure 1.** Proficiency Testing Data Indicating Upward Trend (using control chart software developed in Microsoft access)



\* Note: Z-Score =  $(\text{Individual Lab Result} - \text{Robust Mean}) / \text{Robust Standard Deviation}$ . Preferred Z-Score values are greater than -2 and less than +2.

Date	Method	Mean	Standard Deviation	Lab Result	ASTM Reproducibility	Z-Score	% Recovery	Difference From Mean
<b>Aromatics - D5769</b>								
01/1999		16.02	0.94	15.39	2.04	-0.67	96.1	0.630
02/1999		22.01	1.99	24.2	2.48	1.10	110.0	2.190
03/1999		23.11	1.56	25.5	2.57	1.53	110.3	2.390
04/1999		19.15	1.48	17.67	2.35	-1.00	92.3	1.480
05/1999		20.76	1.15	18.26	2.51	<u>-2.17</u>	88.0	2.500
06/1999		23.34	1.46	21.51	2.78	-1.25	92.2	1.830
07/1999		11.35	1.12	10.06	1.58	-1.15	88.6	1.290
08/1999		15.2	0.59	13.88	1.88	<u>-2.24</u>	91.3	1.320
09/1999		19.74	1.12	17.9	2.41	-1.64	90.7	1.840
10/1999		23.75	1.79	21.23	2.62	-1.41	89.4	2.520
11/1999		18.64	1.26	16.8	2.3	-1.46	90.1	1.840
12/1999		20.14	1.65	17.91	2.32	-1.35	88.9	2.230

Sponsors: ASTM Interlaboratory Crosscheck Program

**Figure 2.** Proficiency Testing Data Indicating Negative Bias (using control chart software developed in Microsoft Access)

## CONTROL CHART TREND ANALYSIS REPORT

Date issued:	TAR No.:
Facility:	Submitted to:
Program:	Test:
Suspicious period (dates):	Period IDs:

Types of Anomalies (check those that apply):

1. Potential Nonconformities

\_\_\_\_\_ Trend – 3 to 5 successive points in an upward (rise) or downward (fall) orientation.

\_\_\_\_\_ Run – 5 to 7 successive points that line up on one side of the mean or center line.

\_\_\_\_\_ Periodicity – a recurring pattern of change in plots in equal intervals.

2. Nonconformities

\_\_\_\_\_ Outliers (a) – two or more single points in succession that fall outside the control or acceptable limit.

\_\_\_\_\_ Outliers (b) – one-third or more of the total number of single points that fall outside the control or acceptable limit.

***To Be Completed By The Laboratory***

Investigation {Describe all actions taken to determine the cause(s) of each anomaly}.

Are in-house control charts available for this test and product?    ☐ yes    ☐ no. If so, please provide copies of the applicable control charts for the suspicious period noted above.

Evaluation (Describe the findings of the investigation and conclusions drawn).

Preventive/Corrective Action (Describe what action was taken to address each anomaly).

Completed by / date:

***To Be Completed By QA Department***

Preventive/Corrective Action Effectiveness (Describe what sample results following the suspicious period were used to evaluate the effectiveness of the preventive/corrective action).

Was the anomaly corrected? If not explain.

Is further action required? If yes, explain.

Closed by:	Date:
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**Figure 3.** Trend Analysis Report

## THE PERFORMANCE-BASED MEASUREMENT SYSTEM (PBMS) AND DATA VERIFICATION AND DATA VALIDATION

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### Abstract

The 21<sup>st</sup> Century is challenging the remediation effort at the Rocky Mountain Arsenal (RMA) into innovative monitoring techniques. The unusual matrices and analytes specific to RMA for which no standard methods are available have necessitated a Performance Based Measurement System (PBMS) for the last ten years. How data verification and data validation should be performed on data generated under PBMS has been an ongoing concern of the regulating community.

PBMS, as stated by the Environmental Protection Agency (EPA), is a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. The regulated entity will be responsible for developing and implementing monitoring programs that demonstrate compliance. This is achieved through specific data quality objectives (DQOs) and quality assurance project plans (QAPPs).

The RVO Quality Assurance Program defines data verification as a non-analytical preliminary review of laboratory data and associated documentation. Data verification ensures that samples are preserved, shipped, maintained, and analyzed in accordance with established data quality requirements, contractual requirements and SOPs. Data validation is a through review of analytical data utilizing laboratory analytical records to assess laboratory performance to quality control criteria, data quality requirements, and procedural requirements.

This presentation will demonstrate how data verification is performed at RMA and the steps a third-party validator can use to assess the usability of data under PBMS.

### Introduction

In 1942 the US Army constructed RMA on the outskirts of Denver, Colorado. The mission of RMA was to produce wartime munitions and chemical weapons. After the end of WWII the Army leased some of its chemical production and storage facilities at RMA to private industry to offset operational cost and maintain the facilities for nation security. In 1952 Shell Chemical Company (now Shell Oil), acquired a pesticide production company at RMA, and continued producing herbicides and agricultural pesticides at RMA for 30 years.

The production of the military chemical weapons, pesticides, insecticides, and herbicides generated many waste streams. Efforts to contain liquid waste began after the discovery in the 1950's that contaminated groundwater caused crop damage. In 1956, the Army constructed Basin F, a 93-acre, asphalt-lined evaporation pond. Basin F held 243 million gallons of liquid waste. Although this disposal method was widely accepted at the time, further contamination resulted.

In June 1996 the Record of Decision (ROD) was signed detailing the remediation efforts at RMA. The cleanup program is managed by the Remediation Venture Office (RVO), a cooperative effort between the U.S. Army, Shell Oil Company, and the United States Fish and Wildlife Service (USF&WS). The United States Environmental Protection Agency (USEPA), the Colorado Department of Public Health and Environment, and the Tri-County Health Department under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), regulate the remediation. Once the remediation is complete, the site will become a permanent part of the National Wildlife Refuge System administered by the USF&WS.

### DISCUSSION

The USEPA has defined PBMS as "a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner."<sup>1</sup> The unusual matrices and analytes routinely found at RMA pose unique problems for the regulator and the analytical laboratories. Analyses for analytes in matrices for which no standard method exists have been required. This has necessitated the modification of existing approved standard methods, thus the



formation of PBMS at RMA. How data verification and data validation should be performed on data generated under PBMS has been an ongoing concern of the regulating community.

The process of generating data of known quality under PBMS at RMA involves three phases:

1. Planning – Defining the data quality objectives (DQOs).
2. Implementation – Writing the quality assurance project plans (QAPPs) and performance-based method standard operating procedures (SOPs).
3. Assessment - Performing data verification and/or data validation.

When any environmental sampling is to be performed at RMA the regulatory agencies along with the RVO meet and identify the DQOs. The DQO process is a planning approach that is used to prepare for data collection activity. It provides a systematic procedure for defining the criteria that a data collection process should satisfy, including when to collect samples, where to collect samples, the tolerable level of decision error for the study and how many samples to collect. Using the DQO process will assure that the type, quantity, and quality of the environmental data used in decision making at RMA will be appropriate for the intended use. The DQO process involves the following steps:

1. Concisely defining the problem,
2. Identifying the decision to be made,
3. Identifying the key inputs to that decision,
4. Defining the boundaries of the study,
5. Developing the decision rule,
6. Specifying tolerable limits on potential decision errors, and
7. Selecting the most resource efficient data collection design.

The QAPP is a formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria. The QAPP must provide sufficient detail to demonstrate that:

1. The project technical and quality objective are identified and agreed upon;
2. The intended measurements, data generation, or data acquisition methods are appropriate for achieving project objectives;
3. The assessment procedures are sufficient for confirming that data of the type and quality needed and expected are obtained; and
4. Any limitations on the use of the data can be identified and documented.

It is recognized that in order to be effective, the QAPP must specify the level or degree of QA and QC activities needed for the particular environmental data operations. Because this will vary according to the purpose and type of work being done a graded approach should be used in planning the work. This means that the QA and QC activities applied to a project will be appropriate with:

1. The purpose of the environmental data operation (e.g., enforcement, research and development, rulemaking);
2. The type of work to be done (e.g., pollutant monitoring, site characterization, risk characterization, bench level proof of concept experiments); and
3. The intended use of the results (e.g., compliance determination, selection of remedial technology, development of environmental regulation).

The RMA Chemical Quality Assurance Plan (CQAP) was developed from the Army Environmental Agency Guidelines and provides the written guidance for operating the RMA QA program. The purpose of the RMA CQAP is:

1. To provide for consistent generation of analytical data;
2. Establish standard practices which permit interlaboratory comparisons of data;
3. Establish procedures for demonstrating that analytical systems are in control; and

4. Ensure that the data produced by the laboratories is not only of highest quality, but scientifically and legally defensible.

Data verification is a non-analytical preliminary review of laboratory data and associated documentation. Data verification ensures that samples were preserved, shipped, maintained, and analyzed in accordance with established DQOs, contractual requirements, and SOPs. The data verification process takes place after the data has been collected. The QAPP will specify different details to which the verification process will be used to confirm that the specific details have been met through compliance, correctness, comparability, and completeness.

A detailed checklist is prepared using each one of these basics of data verification individually or collectively. This checklist should contain everything that will step a person through the verification process. Also, the checklist assures that all data reduction has been correctly performed and that the analytical results reported corresponds to the data acquired and processed.

Completeness verifications on the data packages are performed at RMA ensuring that all data generated has been submitted. The CQAP states that the final data package will be paginated, reviewed, and the validity and usability of data will be attested to in writing in the case narrative and also contain:

1. Approval checklist form(s) showing two levels of review.
2. A page summarizing the data package contents.
3. A narrative summary containing any comments, corrective actions, deviations from SOPs, or other observations on data. If there were no anomalies during the sample run, this fact shall be stated.
4. Data sheets or other printed forms containing reduced final data for all samples and QC samples including the results for blanks, matrix spikes, and LCSs, along with reporting limits for all analytes.
5. Original or copies of field and internal chain of custody (COC) forms.
6. Original or copies of relevant laboratory notebook or logbook pages.
7. Standards preparations, including certificates of analyses, second source information, and solvent purity.
8. Sample preparations and extractions, to include any clean-up documentation.
9. Example calculations.
10. Analytical run sequence.
11. Instrument data reports for tuning reports, if applicable, calibration information, including initial calibration results, daily calibration, and continuing calibration compounds, instrument reports for resolution and degradation checks, if applicable.
12. Chromatograms, strip charts, or other recordings of instrumental output. Include hard copies of before and after chromatograms if manual integrations are performed.
13. Total ion chromatographs of all gas chromatography/mass spectrometer (GC/MS) sample results at or above the reporting limit.
14. The analytical raw data for dilutions must be submitted for the original analysis plus the dilution from which the result was reported.
15. The confirmation results on all non-GC/MS analysis.
16. Copies of corrective action reports.
17. A copy of the electronic data file.

A detailed checklist is constructed using the item required by the RMA CQAP. During the verification process each data each item is checked off to verify that the submitting laboratory has complied with the laboratory approved documents. If during the verification process if any data point(s) has/have not been completed appropriate corrective action(s) must be taken by the laboratory to complete the verification process. Once the data verification process has been completed data validation can be performed.

Data validation is a thorough review of analytical data utilizing laboratory analytical records to assess laboratory performance to quality control criteria, data quality requirements, and procedural requirements. Each project requiring data validation should have a plan developed and defined prior to data validation activities. This plan should address and adequately define at a minimum the following key elements:

1. Level of data validation required based upon the project's DQOs,
2. Data validation schedule,
3. Data validation personnel required,
4. Data validation procedure utilized, and

5. Documentation and reporting requirements,

A determination is made to verify if any federal, state, program, or site-specific data validation criteria have already been established. If for any reason there are not any data validation criteria available a checklist is prepared using the DQOs, the laboratory performance-based SOPs, and the USEPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Review. The laboratory is required to provide QA/QC documentation along with the sample results allowing for the evaluation of the following data quality indicators:

1. Integrity and stability of the samples,
2. Instrument performance during sample analysis,
3. Possibility of sample contamination,
4. Identification and quantitation of analytes,
5. Analytical precision, and
6. Analytical accuracy.

After the determination that all the appropriate and necessary information is available, the data validation procedure commences using the detailed checklist outlining the data quality indicators.

### SUMMARY

PBMS continues to be an on-going process at RMA with the innovative monitoring techniques along with the unusual matrices associated with RMA. Data verification and data validation within the PBMS at RMA provides data, which is of known quality, scientifically sound, and legally defensible

### FOOTNOTES

Federal Register, Vol. 62 No.193, October 6, 1997, Page 52098.

### ACKNOWLEDGEMENTS

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## ETHICS STANDARDS FOR ENVIRONMENTAL AND PETROLEUM TESTING LABORATORIES

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### Abstract

This paper discusses the need for ethics standards in environmental and petroleum testing laboratories and provides guidance on key elements that should be included in an effective ethics program. These elements include implementation of an ethics policy that is strictly enforced; requiring employees to sign an ethics agreement affirming their commitment to ethics and ethical conduct; development and implementation of ethics related policies and procedures; the role of a compliance or ethics officer; ethics assistance and reporting mechanisms; ethics communication and training; and compliance auditing. Examples of typical laboratory problems along with unacceptable solutions and acceptable solutions are presented in order to consider ethical and unethical ways to handle problem situations in the laboratory. By thinking out potentially compromising situations prior to their occurrence, the right choice can be made if and when they do occur.

### Background

Unethical behavior and actions have occurred in environmental and petroleum testing laboratories, despite the existence of laboratory supervision, quality assurance oversight, internal and external audits, and accreditation programs. Unethical conduct, such as intentional alteration of sample or calibration data, can turn into fraud and violation of one or more laws. The government takes fraud very seriously and will take administrative, civil and criminal action against both organizations and individuals that are suspected of committing fraud. Jail time and fines can be the unfortunate long-term consequences of making unethical short-term compromises. New measures are needed to ensure that laboratory employees are educated on the serious consequences of unethical conduct and on the important role of ethics in laboratory testing and data handling.

The need for ethics standards in environmental and petroleum testing laboratories is being recognized and is now included in the requirements for testing. The National Environmental Laboratory Accreditation Conference (NELAC) Standards (Quality Systems, Chapter 5, Section 5.6.2,) requires that ethics training be provided to technical staff at environmental testing laboratories that want to be NELAC accredited. The International Federation of Inspection Agencies (IFIA), Americas Committee, requires that petroleum testing laboratories have an ethics program in order to be a member of IFIA. Guidance is needed for laboratories seeking to implement an ethics program and train their employees in the importance of ethical conduct.

### Introduction

Quality standards in analytical chemistry laboratories are well known and documented. In fact, quality requirements are included in laboratory quality assurance (QA) manuals and analytical methods. Laboratory QA programs include the components necessary to achieve acceptable data and assume that behavior is ethical. However, standards for ethics are frequently not given the same attention and consideration as quality standards. Professional organizations have codes of ethics for their members, e.g., the American Chemical Society, American Society for Quality and American Institute of Chemists. These codes should carry over into work in the laboratory. However, ethical conduct in the chemistry laboratory is either assumed or not discussed. That is, until there is an unethical incident (ethics violation) that brings the need for ethics to the surface. Therefore, laboratories must also have an ethics program to communicate the expected conduct of employees, and describe what constitutes unethical behavior.

Ethics is a code of right and wrong that dictates personal and professional conduct. Ethical behavior is behavior that conforms to accepted professional standards of conduct; unethical behavior therefore is behavior not conforming to those standards. Fraud is an intentional act of deceit that may result in legal prosecution. Traditional laboratory QA programs have not adequately addressed ethics related matters because: 1) QA programs were not intended to address ethical or unethical behavior (they assume that behavior is ethical); 2) the scope of ethics transcends the matter of quality; and 3) QA programs deal with group activities rather than individual behavior. The decision to act ethically or unethically is an individual decision, not a group decision (although there have been instances where a group of employees acted unethically, it still was the individual's decision to act unethically.) Even the best laboratory QA program cannot ensure that employees will act ethically. Further, laboratory QA programs are not always effective in ensuring method and project compliance.

The unfortunate occurrences of unethical behavior in the laboratory community resulting in fraud are most likely due to either: 1) lack of ethics education and awareness prior to the fraudulent act (if analysts knew what could happen to them if they make unacceptable compromises, they would certainly not do it); or 2) lack of knowledge of confidence in appropriate ways to handle non-compliant data and problem situations. Unethical conduct in the laboratory does not generally occur when everything is going smoothly, it occurs when there are problems and pressure to achieve difficult or unrealistic objectives. The analytical community needs to do a better job collectively in educating laboratory analysts and technicians on the role of ethics in their work and in better ways to handle problem situations. Most individuals do not personally gain from committing an unethical act except to relieve some pressure that they feel, whether it is real or perceived. The impact of unethical behavior and fraud has been devastating to laboratories and laboratory employees, as well as to data users that must repeat entire projects if the original data is determined to be non-authentic. To better ensure that laboratory staff act ethically, an effective ethics program must be implemented in conjunction with the laboratory QA program.

### **Relevant Criminal Laws**

An unethical action becomes a fraudulent act when the law is violated. For example, it is unethical if an analyst intentionally changes the instrument clock to make samples appear to be analyzed within holding time, when in fact they were not. It is unethical to intentionally manipulate instrument calibration or QC data to make the calibration or QC analysis meet an acceptance limit, when in fact the actual data were not acceptable. It is also unethical to intentionally alter sample data so that results appear to be "on-spec" when in fact the results were "off-spec". An unethical act turns into a fraudulent act when falsified data is faxed or mailed to the client or other party. Faxing or mailing false information is an example of a violation under the laws relating to wire fraud or mail fraud, respectively. The sender could be charged with wire fraud or mail fraud, as well as making false statements if the work was done under a government contract. Relevant criminal laws that apply are as follows: False Claims – 18 U.S.C. § 287; False Statements – 18 U.S.C. § 1001; Mail Fraud – 18 U.S.C. § 1341; Wire Fraud – 18 U.S.C. § 1343; Conspiracy – 18 U.S.C. § 371; and Misprision (Concealment) of Felony – 18 U.S.C. § 4. Violations of these laws can result in substantial fines and imprisonment for up to five years. Press releases on laboratory investigations and convictions demonstrate that multiple charges of fraud are filed against laboratories and personnel that report false information.

Ethics violations and fraud affect both individuals and organizations (private and public). Regulatory agencies (i.e., the U.S. Environmental Protection Agency) and law enforcement officials (i.e., State attorneys) aggressively pursue and prosecute both individuals and organizations found to be in violation of the law. Enforcement actions are increasing as well as the severity of penalties for environmental crimes. Companies can face three types of legal action if they break the law: 1) administrative action, 2) civil action and 3) criminal action. Administrative action can result in debarment or probation for five or more years. Civil action can result in large fines of up to several million dollars. Criminal action can result in prison sentences for business owners or management officials. All of these actions can seriously damage the reputation of a company, cause a loss of revenue and customer business, and result in shutdown of the affected office(s) of the company. Further, attorney costs can run in the hundreds of thousands of dollars to represent and defend an organization charged with fraud, regardless of the final outcome.

An individual who commits an unethical act and/or breaks the law can face serious disciplinary action up to and including termination. Civil and criminal action can be taken against the individual, resulting in large fines and prison and/or probation sentences. Company lawyers may not provide legal assistance to an individual who commits an unethical act that results in a fraud charge(s); the individual must then seek and pay for his own legal assistance which can be very expensive. Further, negative exposure hurts the individual's chances of ever getting a job in his field again. The moral of this story is that short-term compromises are never worth the long-term consequences. Ethical conduct is the best course of action.

### **Implementing an Ethics Program**

*Ethics References.* Legal guidance documents and web sites are sources for ethics information, although they may not specifically relate to laboratory activities. Two recent EPA documents provide guidance on the deterrence and detection of laboratory fraud.<sup>1-2</sup> Related information is provided in other publications on data authenticity, compliance and ethics.<sup>3-8</sup>

*Ethics Policy or Statement.* An ethics program must have an ethics policy or statement. This policy or statement should define the company or organization's position on ethics and state what is expected of its employees or members with regards to ethical behavior. For example, a company's ethics policy may include the following

items: "All employees at all times shall conduct themselves and the business of the Company in an honest and ethical manner. Compliance with this policy shall be strictly enforced." The ethics policy should be documented and posted for all employees to view.

*Employee Ethics Agreements.* Employee commitment to comply with the ethics policy should be affirmed and documented on an Employee Ethics Agreement that each employee must sign as a condition of his or her employment. These agreements may be updated as needed and signed each year to reaffirm each employee's understanding of, and commitment to, ethical behavior. If an employee is found to be in violation of the company Ethics Policy or the signed Employee Ethics Agreement, the employee may be terminated immediately.

*Ethics Communication.* Ethics should be communicated often: verbally, in writing, and by example. Laboratory staff should consider and discuss ethics, addressing any questions to the appropriate parties. Supervisors and managers should be readily available to assist employees in managing problem situations (to prevent ethics violations), and they should act ethically at all times to set a good example for their employees. Corporate management should frequently discuss their commitment to ethics with their managers and employees. Ethics should be discussed at meetings and other opportunities where employees are present. Videos may be developed by corporate management to further communicate ethics to company employees. There are many opportunities to include ethics in writing. Ethics posters are one form of communication. Ethics standards and reference to appropriate ways of handling non-compliant data and problem situations should be included in the QA manuals and standard operating procedures. Reference to ethical behavior should be included in contracts, sub-contracts, employment applications, and project plans. Ethics information and questions on ethics knowledge can be included in training records.

*Ethics Program Management.* The ethics program should be managed by a senior management employee with the authority, skills and availability to perform such an assignment. The ethics program manager should report to upper management on a regular basis on the status of ethics activities within the organization. Companies and other organizations may also elect to form an Ethics Committee with members from their upper management staff or Board of Directors that meet on a regular basis to set ethics policy and discuss ethics related matters.

*Ethics Procedures.* Policies and procedures for ethical conduct and for reporting and investigating suspected ethics violations should be developed and included in the organization's policy and procedures manual. An ethics procedure should define ethical conduct and what constitutes unethical behavior and how it is handled. Disciplinary action for ethics violations, up to and including termination, should be stated in the ethics procedure. Fair procedures for reporting and investigating alleged unethical behavior should be included in an ethics reporting and investigation procedure. Ethics procedures as well as other company procedures should be accessible to all employees. The application of these procedures for handling suspected or actual ethics violations should be uniform and consistent for all employees.

*Zero Tolerance Policy.* Organizations should have a zero tolerance policy on unethical activities, scientific misconduct and intentional lack of compliance with required procedures. Unethical behavior would include intentional falsification of data or records, such as professional credentials, employment records, time sheets, sampling or sample handling records, laboratory worksheets or logbooks, instrument settings or data, sample results or data, and laboratory analysis reports. Intentional lack of compliance or deliberate lack of adherence to company and method requirements would apply to an employee that purposely did not follow required procedures for instrument calibration, quality control, standards and reagents preparation, sample handling, sample preparation and analysis, or data processing and reporting.

Laboratories may wish to go one step further and define specific actions in the laboratory that are unacceptable or unethical. This may be in the form of a policy that employees are required to sign as demonstration of their understanding and commitment to comply with it. While most laboratory procedures define what employees are required to do, this policy ensures that employees are educated as to what they are not allowed to do. Refer to Table 1 for examples of typical laboratory problems and both unacceptable and acceptable ways to handle each situation. Laboratories that are proactive in informing employees of what constitutes unethical behavior have a much better chance of preventing fraud than laboratories that do not.

*Ethics Assistance and Reporting Mechanism.* Organizations should have a single point of contact for assisting employees with questions on ethics related matters and for reporting observations of suspected unethical behavior or business conduct. A "helpline" or "hotline" is such a mechanism where phone calls, faxes or other

correspondence on ethics concerns, questions or reports of suspected unethical behavior can be directed and then addressed appropriately. The phone numbers and addresses for the helpline or hotline should be documented and readily available to all employees. The helpline or hotline can be manned by a senior management employee, such as the compliance program manager, or by an outside service. All inquiries should be acted upon in a prompt matter according to appropriate procedures.

**Compliance Plan.** A compliance plan is all of the procedures used for ensuring compliance with company, client and regulatory requirements. The compliance plan should include or refer to policies and procedures on business conduct, especially ethics. Also include or refer to technical and quality assurance procedures used by the laboratory and required by client, method or regulatory agencies to ensure that data are accurate and traceable. The compliance plan should further include or refer to environmental management activities and procedures used for chemical and waste handling to comply with federal, state and local regulations. A compliance plan may also include a quality management program such as ISO 9002, and quality standards for laboratories such as ISO Guide 25 (to become ISO 17025) or the NELAC quality system standards.

**Ethics Training.** Ethics training should be provided to all employees and include, at a minimum, training on the organization's ethics policy and procedures. Ethics training should communicate the organization's expectations on ethical conduct and include examples of unethical activities and their impact (i.e., civil and criminal penalties) to demonstrate that short-term compromises are not worth the long-term consequences. Questions or tests to verify understanding of the ethics requirements should be part of the training course. Ethics training should be documented on training forms and included in the employee training or personnel files. Training on laboratory procedures should be ongoing and based on each individual and their work assignments. Additional training on solving problems and managing work loads is critical to assist analysts in proper preventive/corrective action on analytical problems and the use of appropriate techniques for achieving desired productivity goals.

**Compliance Audits.** Adherence to the compliance plan, ethics program and associated procedures/requirements should be checked on a regular basis via compliance audits. The compliance officer, quality assurance staff or outside consultants may conduct compliance audits to determine if the ethics policy and procedures are being followed as well as technical and environmental management procedures. Any findings of non-compliance with company, client or government requirements should be documented and provided to company management. Immediate and appropriate action should be taken on any serious findings, up to and including issuance of a Stop Work Order on the affected areas. Prompt and effective preventive/corrective action should be taken on all findings and reported back to the auditing body for review and approval with copies provided to management. Verification of preventive/corrective action implementation should be performed in a timely manner by the auditors to ensure that preventive/corrective action was complete and effective in addressing the audit findings. Any unresolved items should be reported to management for immediate action.

With ethics as an established goal, organizations should further strive to find ways to monitor and bench mark the ethical behavior of their employees.

## **Conclusion**

Ethical conduct in the laboratory is not guaranteed by the sole reliance on laboratory QA programs that were not designed to address ethical matters. In spite of the existence of good laboratory QA programs, unethical practices have occurred in environmental and petroleum testing laboratories and data quality has suffered. A new approach is needed to ensure that ethics and ethical behavior is a foundation for the performance of all work in the laboratory. Ethics must be built individually and collectively into a laboratory organization. Each laboratory employee, including managers, must understand and commit to the code and perform his or her work in an ethical manner. Ethics awareness and the implementation of a comprehensive ethics program in analytical chemistry laboratories can help to ensure better data quality and prevent further unethical acts from occurring, thereby sparing any more laboratories, laboratory staff or clients from suffering the serious consequences of fraud. A comprehensive ethics program, based on the guidance provided in this article, in conjunction with an effective

**Table 1.** Typical Laboratory Problems and Unacceptable and Acceptable Solutions

Problem	Unacceptable Solution	Acceptable Solution
Lack of time or resources to perform testing	<b>Making up Data (Dry Labbing) or Other Information</b> – Creating data for an analysis that was not performed or creating information that is not true.	Analytical results for all samples and quality control (QC) must be based on actual analyses that were performed. Documented data must match actual data. Sampling information must be based on actual sampling events.
Holding time near or past	<b>Improper Clock Setting (Time Traveling) or Improper Date/Time Recording</b> – Resetting the internal clock on an instrument to make it appear that a sample(s) was analyzed within a specified holding time when in fact it was not. Alternately, changing the actual time or recording a false time to make it appear that holding times were met, or changing the times for sample collection, extractions or other steps to make it appear that they were performed at the correct time when in fact they were not.	The recorded date and time of collection, preparation or analysis must match the actual date and time that the action was performed. Documented dates and times must represent actual dates and times. Samples exceeding holding times must be reported as such; a case narrative is recommended.
DFTPP or BFB not meeting acceptance criteria	<b>Improper GC/MS Tuning</b> – Artificially manipulating GC/MS tuning data to produce an ion abundance result that appears to meet specific QC criteria when in fact the criteria were not met.	GC/MS tuning data must be generated and reported according to proper techniques without manipulation to the peak or mass spectrum. Preventive/corrective action must be taken on data not meeting required criteria.
Calibration or QC data not meeting acceptance criteria	<b>Improper Peak Integration (Peak Shaving or Enhancing)</b> – Artificially subtracting or adding peak area to produce an erroneous area that forces data to meet specific QC criteria when in fact the criteria were not met.	Instrument peaks must be consistently integrated and reported according to proper techniques, generally baseline to baseline, valley to valley or a combination of the two. Peak area cannot be subtracted or added to force data to meet specified criteria. Preventive/ corrective action must be taken on instrument data not meeting required criteria.
Calibration or QC data not meeting acceptance criteria	<b>Improper Calibration/QC Analysis</b> – a. Performing multiple (more than two) calibrations or QC runs (including calibration verifications, LCSs, spikes, duplicates and blanks) until one analysis barely meets criteria, rather than taking needed preventive/corrective action after the second failed analysis, and not documenting or retaining data for the other unacceptable data. b. Using the incorrect (previous) initial calibration to make calibration verification data appear to be acceptable when in fact it was not acceptable when compared to the correct initial calibration. c. Discarding points in the initial calibration to force the calibration to meet an acceptance criteria. d. Discarding points from an MDL study to force the calculated MDL to be higher or lower than the actual value.	a. All calibration and QC data associated with sample analyses must be documented. Preventive/ corrective action must be taken and documented if calibration and/or other QC criteria are not met. b. Acceptance of calibration verification data must be based on the correct initial calibration. c. Calibration points can only be rejected for inclusion in the calibration curve if a known error was made or if a statistical evaluation indicates that a point can be discarded. When multiple target analytes are included in each calibration standard, it may be necessary to discard selected upper or lower points for individual target analytes. Points can be discarded at the upper end of the curve if the linear range of the detector has been exceeded. For these cases, dilute samples that exceed the highest point of the calibration curve. Points can be discarded at the lower end of the curve if the detector is not producing a response. For these cases, the laboratory-reporting limit must be adjusted accordingly. d. Data points for MDL studies can only be rejected for inclusion in the MDL calculation if a known error was made or if a statistical evaluation indicates that a point can be discarded.



**Table 1.** Typical Laboratory Problems and Unacceptable and Acceptable Solutions (cont.)

<b>Problem</b>	<b>Unacceptable Solution</b>	<b>Acceptable Solution</b>
QC samples or spikes not meeting acceptance criteria	<p><b>Misrepresentation of QC Samples and Spikes</b> – Misrepresenting QC samples or spikes as being digested or extracted when in fact they were not actually digested or extracted. For example:</p> <ul style="list-style-type: none"> <li>a. Adding surrogates after sample extraction rather than prior to sample extraction.</li> <li>b. Reporting post-digested spikes or duplicates as pre-digested spikes or duplicates.</li> <li>c. Not preparing or analyzing method blanks and laboratory control samples (LCSs) the same way that samples are prepared or analyzed in order to make it appear that method blank or LCS results are acceptable when in fact they may not be.</li> </ul>	<p>QC samples and spikes must be prepared, analyzed and reported according to appropriate procedures.</p> <ul style="list-style-type: none"> <li>a. Surrogates must be added prior to sample extraction.</li> <li>b. Post-digestion spikes and duplicates must be reported as post-digested and must not be misrepresented as pre-digestion spikes and duplicates.</li> <li>c. Method blanks and LCSs must be prepared and analyzed the same way that samples are prepared and analyzed.</li> </ul> <p>Any QC results outside of acceptance criteria must be reported as such; a case narrative is recommended.</p>
Calibration or QC data not meeting acceptance criteria	<p><b>File Substitution</b> – Substituting previously generated files (runs) for a non-compliant calibration or QC run to make it appear that an acceptable run was performed when in fact it was not.</p>	<p>All data must be generated and reported for actual analyses performed. Reported dates and times for all analyses must match actual dates and times. Substitution of files is not permitted.</p>
Calibration or QC data not meeting acceptance criteria	<p><b>Unwarranted Manipulation of Computer Software</b> – Unwarranted manipulation of computer software to force calibration or QC data to meet criteria, and removing computer operational codes, such as “M” flag.</p>	<p>Computer manipulation is allowed only for warranted reasons and any manipulation should be minimal and traceable. Removal of computer operational codes is not permitted.</p>
Analytical conditions for standard do not work for sample	<p><b>Improper Alteration of Analytical Conditions</b> – Improperly altering analytical conditions, such as changing the instrument conditions for sample analyses from those used for standard analyses. Also using different procedures to process sample data than those used for standards.</p>	<p>All sample analyses must be performed under the same conditions as those used for standard analyses. Any alterations of analytical conditions must be allowable under the method requirements. All sample data must be processed by the same procedures as those used for processing standard data. Any discrepancies must be documented</p>
Sample not analyzed at appropriate level or not reported at correct detection limit	<p><b>Overdilution of Samples or Misrepresentation of Detection Limits</b> – Intentionally diluting a sample to such an extent that no analytes (target or non-target) are detected without justification as to why the high dilution was made. Reporting a detection limit that does not represent the sample analysis (e.g., not including dilution factor in sample detection limit)</p>	<p>Dilutions must be made on a reasonable basis, such as high concentrations of target or non-target analytes, matrix interferences, oily samples, and other components in the sample that could harm the instrument. Include details on the reason for the dilution in a case narrative. Sample detection or reporting limits must include and dilution factors.</p>
Non-compliant data	<p><b>Deletion of Non-Compliant Data</b> – Intentional deletion or non-recording of non-compliant data to conceal the fact that analyses were non-compliant.</p>	<p>All data associated with sample collection and analysis, including any out of control events or non-compliant data, must be documented and retained. Preventive/ corrective action must be taken and documented for any non-compliant data.</p>
Undesirable situation with analysis or sample; knowledge of unethical conduct	<p><b>Concealment of a Known Problem</b> – Concealing a known analytical or sample problem from laboratory management and/or client. Concealing a known unethical behavior or action from laboratory or corporate management.</p>	<p>Any knowledge of analytical or sample problems must be communicated to laboratory management and the client. Any knowledge of unethical behavior or actions must be fully communicated to laboratory or corporate management.</p>

laboratory QA program, will provide a strong foundation for ethical conduct and improved data quality.

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## **AUTOMATED DATA REVIEW (ADR), CONTRACT COMPLIANCE SCREENING (CCS), AND ENVIRONMENTAL DATABASE MANAGEMENT SYSTEM (EDMS) SOFTWARE APPLICATIONS FOR THE SACRAMENTO DISTRICT FORT ORD PROJECT**

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## Abstract

This poster presentation is an overview of the Contract Compliance Screening (CCS), Automated Data Review (ADR), and Environmental Database Management System (EDMS) software programs developed by Laboratory Data Consultants, Inc. under contract to the Army Corps of Engineers, Sacramento District for the Fort Ord RI/FS project. The software programs use an electronic data deliverable (EDD) format based upon data elements originally documented in the Implementation Guide for the Department of Energy Environmental Management Electronic Data Deliverable Master Specification (DEEMS). The software was developed on a Microsoft ACCESS 97 platform. Customized modules perform automated data review (EPA Level 3) and provide the user with discrete data qualification. The qualified data is exported into a master database for overall project use.

The EDD format includes QA/QC batch links and routine accuracy and precision parameters such as surrogate, matrix spike, and laboratory control sample recoveries. In addition, initial and continuing calibration and GC/MS tuning data are delivered in this format. Development of the EDD integrated these data elements required by end users with consideration for the current data deliverable capabilities of commercial laboratories.

The CCS software verifies the completeness and compliance with the EDD format. The software references a project specific library built upon the QAPP in verifying compliance and completeness. EDD deficiencies are detailed in an outlier report. Access to the EDD file in table format allows for quick and easy correction of errors. The ADR software is initiated by the data user (i.e., Army Corps chemist, prime contractor, etc.) to review analytical data based upon project specific criteria. Upon execution of the program, data is qualified using

standard Army Corps/EPA data flags and exported into a master database. Command buttons generate reports such as a rejected data table, method blank contamination, surrogate outliers, etc. Forms and view screens also provided on-line review of data qualifiers.

The Environmental Database Management System (EDMS) compiles the validated data downloaded from the ADR system. The database program has user functions which allows for comparison of primary data versus QA split lab data, comparison of results against project action limits or PRGs/MCLs, and calculates the completeness values for each test over any period of time. The four types of completeness values include contract, analytical, technical, and field sampling completeness.

In conclusion, the CCS, ADR, and EDMS software programs were developed as tools to support technical staff in the evaluation of analytical chemistry data using an expedited and cost effective automated process. The EDD provides a standardized format. This format allows for streamlining at the laboratories to produce deliverables which can be verified immediately for completeness and compliance against project criteria using the CCS software. The EDMS allows the data end user to efficiently evaluate large data sets for key indicators and ultimately determine the usability of the data.

### **Introduction**

This poster presentation is an overview of the Contract Compliance Screening (CCS), Automated Data Review (ADR), and Environmental Database Management System (EDMS) software programs developed by Laboratory Data Consultants, Inc. under contract to the Army Corps of Engineers, Sacramento District for the Fort Ord RI/FS project. The software programs use an electronic data deliverable (EDD) format based upon data elements originally documented in the Implementation Guide for the Department of Energy Environmental Management Electronic Data Deliverable Master Specification (DEEMS). The software was developed on a Microsoft ACCESS 97 platform. Customized modules perform automated data review (EPA Level 3) and provide the user with discrete data qualification. The qualified data is exported into a master database for overall project use.

### **Summary**

The Electronic Data Deliverable (EDD) format is divided into a three table relational structure. The tables are linked with selected key fields. The tables are divided into a Results Table, a Sample Table, and an Instrument Calibration Table. These files include QA/QC batch links and routine accuracy and precision parameters such as surrogate, matrix spike, and laboratory control sample recoveries and initial and continuing calibration and GC/MS tuning data. Development of the EDD integrated these data elements required by end users with consideration for the current data deliverable capabilities of commercial laboratories. The following is the list of field names in the three tables.

The CCS software verifies the completeness and compliance with the EDD format. The software references a project specific library built upon the Quality Assurance Project Plan (QAPP) in verifying compliance and completeness. EDD deficiencies are detailed in an outlier report. Access to the EDD file in table format allows for quick and easy correction of errors.

The ADR software is initiated by the data user (i.e., Army Corps chemist, prime contractor, etc.) to review analytical data based upon project specific criteria. Upon execution of the program, data is qualified using standard Army Corps/EPA data flags and exported into a master database. Command buttons generate reports such as a rejected data table, method blank contamination, surrogate outliers, etc. Forms and view screens also provided on-line review of data qualifiers. See Attachment 1 for an example of user screens.

The Environmental Database Management System (EDMS) compiles the validated data downloaded from the ADR system. The database program has user functions which allows for comparison of primary data versus QA split lab data, comparison of results against project action limits or PRGs/MCLs, and calculates the completeness values for each test over any period of time. The four types of completeness values include contract, analytical, technical, and field sampling completeness. See Attachment 2 and 3 for an example of user screens.

In summary, the CCS, ADR, and EDMS software programs were developed as tools to support technical staff in the evaluation of analytical chemistry data using an expedited and cost effective automated process. The EDD provides a standardized format. This format allows for streamlining at the laboratories to produce deliverables which can be verified immediately for completeness and compliance against project criteria using the CCS software. The EDMS allows the data end user to efficiently evaluate large data sets for key indicators and ultimately determine the usability of the data.

Results Table (A1)	Instrument Table (A2)	Sample Analysis Table (A3)
Client_Sample_ID	Instrument_ID	Project_Number
Lab_Analysis_Ref_Method_ID	QC_Type	Project_Name
Analysis_Type	Analyzed	Client_Sample_ID
Lab_Sample_ID	Alternate_Lab_Analysis_ID	Collected
Lab_ID	Lab_Analysis_ID	Matrix_ID
Client_Analyte_ID	Lab_Analysis_Ref_Method_ID	Lab_Sample_ID
Analyte_Name	Client_Analyte_ID	QC_Type
Result	Analyte_Name	Shipping_Batch_ID
Result_Units	Run_Batch	Temperature
Lab_Qualifiers	Analysis_Batch	Lab_Analysis_Ref_Method_ID
Detection_Limit	Lab_Reporting_Batch	Preparation_Type
Detection_Limit_Type	Relative_Percent_Standard_Deviation	Analysis_Type
Retention_Time	Percent_Difference	Prepared
Analyte_Type	Several fields for BFB/DFTPP ratios and peak ID	Lab_ID
Percent_Recovery		QC_Level
Relative_Percent_Difference		Result_Basis
Reporting_Limit		Total_Or_Dissolved
Reporting_Limit_Type		Dilution
Reportable_Result		Handling_Type
		Handling_Batch
		Leachate_Date
		Percent_Moisture
		Method_Batch
		Preparation_Batch
		Run_Batch
		Analysis_Batch
		Lab_Reporting_Batch
		Lab_Receipt
		Lab_Reported

## Attachment 1

QC Outlier Report: Continuing Calibration Verification							
Analysis Batch : msv0209249a		Analysis Method : 8260B		Analysis Date : 09/24/1999			
Lab Reporting Batch : P909410				Lab ID: SAL-Pet			
Analyte Name	Calibration Result		Project Limits				
	Relative Response Factor	Percent Difference	Relative Response Factor	Positive Percent Difference		Negative Percent Difference	
				Lower Limit	Upper Limit	Lower Limit	Upper Limit
Chloroethane	0.0294	12.7	0.05	25.0		-50.0	-25.0
Dibromochloromethane	0.295	-27.7		25.0		-50.0	-25.0

Associated Samples	
Client Sample ID	Lab Sample ID
9937E012223F	P909410-24
9937E012224F	P909410-25
9937E012225F	P909410-26
9937M018102F	P909410-19
9937M018103F	P909410-20
9937M018104F	P909410-21
9937M022105F	P909410-22

**Figure 1.** Quality Control Outlier Report — The ADR program has built in QC outlier reports which summarizes calibration deficiencies using the project specific method and validation criteria.

Data Qualification Report ( All Qualifiers )																		
Client Sample ID : 9937E017233F				Lab Report Batch : P909410				Lab ID : SAL-Pet										
Sample Date : 09/15/1999				Analysis Type: RES				Sample Matrix : AQ										
Lab Sample ID: P909410-14																		
Validated By / Date : _____				Approved By / Date : _____														
Analyte Name	Result	Result Units	Lab Qual	Rep Res	Overall Val Qual	Temp	HT	MB	LCS	MS	Lab Dup	Surr	Rep Limit	Field QC	Tune	IC	ICV	CV / CCV
Analysis Method : 6010B																		
Potassium	3000	ug/l	J	YES	J+					J+								
Analysis Method : 8260B																		
Bromodichloromethane	0.0709	ug/l	J	YES	J+							J+						
Chloroform	0.0750	ug/l	J	YES	UJ							J+		U				
Dibromochloromethane	0.0660	ug/l	J	YES	J+							J+						
Freon 113	0.222	ug/l	J	YES	J+							J+						
Methylene chloride	0.167	ug/l	J	YES	UJ							J+		U				
Trichloroethane	0.162	ug/l	J	YES	J+							J+						

**Figure 2.** Data Qualification Summary Report — The ADR program provides a summary of qualified data itemizing each quality control area.

## Attachment 2

**Comparison of Field Sample Results versus PRG and MCL Data**

**Comparison Options**

☒ 1. By Project, Sampling Event, SDG, and/or Method

☐ 2. By Sample ID for Projects selected in Option 1

**Sampling Events**

Beginning Date:  Ending Date:  Annual:  "mm/dd/yyyy"

**Option 1: Choose Projects to process**

Project No.	Project Name / Description	Report Tag
36086-010213	Fort Ord Qly Groundwater Monitoring	<input checked="" type="checkbox"/>
36273-001123	Fort Ord Qly Groundwater Monitoring	<input checked="" type="checkbox"/>

Select All Clear All

**Choose Lab Reporting Batch and Analytical Method**

Lab Reporting Batch	Report Tag	Lab Method ID	Report Tag
P906197	<input checked="" type="checkbox"/>	160.1	<input checked="" type="checkbox"/>
P906309	<input checked="" type="checkbox"/>	300.0	<input checked="" type="checkbox"/>
P906373	<input checked="" type="checkbox"/>	310.1	<input checked="" type="checkbox"/>
P906391	<input checked="" type="checkbox"/>	325.3	<input checked="" type="checkbox"/>
P906399	<input checked="" type="checkbox"/>	340.2	<input checked="" type="checkbox"/>
P906511	<input checked="" type="checkbox"/>	353.2	<input checked="" type="checkbox"/>
P906592	<input checked="" type="checkbox"/>	365.2	<input checked="" type="checkbox"/>
P909122	<input checked="" type="checkbox"/>	375.4	<input checked="" type="checkbox"/>
P909139	<input checked="" type="checkbox"/>	394.2	<input checked="" type="checkbox"/>
P909307	<input checked="" type="checkbox"/>	501.08	<input checked="" type="checkbox"/>

Select All Clear All

**Choose one or more Contamination Limits from the list**

Category and Description	Report Tag
Ambient Air	PRG value <input checked="" type="checkbox"/>
Industrial Soil	PRG value <input checked="" type="checkbox"/>
MCL Action Level	California Title 22 Maximum Concentration Limit <input checked="" type="checkbox"/>
MCL Primary	California Title 22 Maximum Concentration Limit <input checked="" type="checkbox"/>
MCL Secondary	California Title 22 Maximum Concentration Limit <input checked="" type="checkbox"/>
Residential Soil	PRG value <input checked="" type="checkbox"/>
STLC	California Title 22 Soluble Threshold Limit Conc <input checked="" type="checkbox"/>

Clear All

FortOrd: Fort Ord Monitoring Wells at OUs 1 and 2

Generate Cont Limits Comparison View Results Cont Limits Comparison Run Contamination Limits Comparison Printout Menu

Save/Archive: Greater than Limit Spreadsheet: All Analyses 1001200406ContaminationLimits

Laboratory Data Consultants, Inc.

**Figure 3.** Comparison of Analytical Results vs PRGs and MCLs - The EDMS application provides user tools to allow for the comparison of field sample results against project specific PRGs and MCLs. The data can be filtered and selected based on many unique criteria.

## Attachment 3

**Results of Laboratory QA Split Data Comparison**

Lab QA Split Validation flag	Lab QA Split Date/TIME	Sample Matrix	Analytical Method	Analysis Name	Client Sample ID	Sample Date/Time	Lab ID	Lab Unit	Result	Detection Limit	Reporting Limit
Outlier	147.93	AQ	82600	Isomers 1,2 Dichlorobenzene							
					Primary Sample	06/07/1999 0:00	SAL-Pet	1	0.100	0.0800	0.500
					QA Split Sample	06/07/1999 0:00	NCL	1	0.2	0.0800	0.500
	7.85	AQ	82600	Trichlorobenzene							
					Primary Sample	06/07/1999 0:00	SAL-Pet	1	41.2	0.0500	0.500
					QA Split Sample	06/07/1999 0:00	NCL	1	44.2	0.0500	0.500
Outlier	191.49	AQ	82600	Vinyl chloride							
					Primary Sample	06/07/1999 0:00	SAL-Pet	1	0.0690	0.0400	0.500
					QA Split Sample	06/07/1999 0:00	NCL	1	0.5	0.0400	0.500
Outlier	168.13	AQ	160.1	Total Dissolved Solids							
					Primary Sample	06/07/1999 0:00	SAL-Pet	1	300	5.00	5.00
					QA Split Sample	06/07/1999 0:00	NCL	1	3500	5.00	5.00
Outlier	52.87	AQ	310.1	Bicarbonate Alkalinity							
					Primary Sample	06/07/1999 0:00	SAL-Pet	1	64.0	20.0	20.0
					QA Split Sample	06/07/1999 0:00	NCL	1	112	20.0	20.0
Outlier	54.55	AQ	310.1	Total Alkalinity							
					Primary Sample	06/07/1999 0:00	SAL-Pet	1	64.0	20.0	20.0

Database Name/Description: FortOrd: Fort Ord Monitoring Wells at OUs 1 and 2

Printout Menu

**Figure 4.** Laboratory QA Split Sample Comparison — The EDMS program allows the user to compare QA split sample results generated from two different laboratories.



## **FASTAC-CLP**

Charles Appleby

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A well known saying about analytical data gathering goes like this, "Fast, accurate, cheap: pick any two." It is not a new phenomenon to find EPA project managers who are willing to choose fast and cheap at the expense of accuracy. In response to this, and with the support of its contractors, Region 4 EPA has worked to make the contract laboratory program (CLP) more attractive to its project managers. This has been done by incorporating tools provided by EPA headquarters, such as Field and Analytical Services Teaming Advisory Committee - CLP (FASTAC-CLP), Computer-Aided Data Review and Evaluation (CADRE), and Data Assessment Tools (DAT), into its quality assurance program. We will discuss how this system allowed us to cut the time required for data validation in half while maintaining a high and known level of quality.

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## **LAB PROCEDURES FOR ANALYST DEMONSTRATION OF METHOD PROFICIENCY**

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### **ABSTRACT**

Prior to laboratory analysis using a respective methodology, the Lockheed Martin Region 6 ESAT team demonstrates the analyst's proficiency and the capability of using laboratory instrumentation to perform the analysis, as specified in the referenced method and the performance criteria. Each analyst must individually demonstrate their proficiency with a particular laboratory technique through an initial demonstration of proficiency prior to conducting the analysis on laboratory samples. Continuing capability is ensured through the analysis of performance evaluation samples, method detection limit studies, and continuing demonstration of ability studies, usually on an annual basis. These activities are documented as part of the overall Lockheed Martin ESAT Quality Assurance (QA) program requirements.

### **INTRODUCTION**

Laboratories are required to provide clients with accurate, defensible results in a timely and cost-effective manner. The release of reliable data is important as the laboratory is a substantial link in the decision making process involving site assessments, ongoing remediations, and public health issues.

We would like to present the procedures we have developed for our group to ensure that our analysts are capable and qualified in performing analytical testing methods for our clients. Our program involves taking an analyst through a development and training course that includes an initial demonstration of proficiency for organic extraction and analysis, inorganic digestion and analysis, and performance evaluation (PE) sample analysis. Our analyst training also covers method detection limits (MDLs), instrument detection limits (IDLs), calculations, control charts, data reduction, proper integration techniques, data validation, and data integrity. This program is utilized for new employees and on a continuing basis for senior analysts.

We have developed these requirements based on EPA SW 846 methods, National Environmental Laboratory Accreditation Conference (NELAC) standards, and the CLP Statement of Work (SOW), and modified them to cover both organic and inorganic laboratory procedures.

### **INITIAL DEMONSTRATION OF PROFICIENCY**

The requirement for an analyst's proficiency to perform a specific method is demonstrated through (1) instrument calibration, (2) method blank evaluation, (3) the analysis of four mid-level standards within method precision and

accuracy requirements, (4) the completion of a method detection limit (MDL) study, and (5) the analysis of a single-blind PE sample containing at least 10 percent of the method analytes.

Additionally, upon acquiring new instrumentation, such as a mercury analyzer to be used for an existing procedure, an initial demonstration of instrument capability must be completed. This is to establish that the new instrument is capable of generating acceptable data prior to its use in the analysis of environmental samples. Any time an analytical system is significantly altered, its capability must also be demonstrated again.

In addition to demonstrating the capability to properly operate a laboratory instrument, each individual analyst must demonstrate proficiency with an analytical technique before generating data for environmental samples. Analytical techniques are defined by the technology employed and should be approached similarly. Some examples are listed below.

1. The extraction of aqueous samples for pesticide analysis may be performed using either solid phase extraction or continuous liquid-liquid extraction. As these are grossly different extraction techniques, the analyst should complete two initial demonstrations of proficiency prior to extracting samples using either of the two techniques.
2. The extraction of aqueous samples for semi-volatile GC/MS analysis using continuous liquid-liquid extraction employs the same techniques as those used for pesticides extractions, so an additional demonstration of extraction proficiency would not be required.
3. Instrumentation techniques are separated based on the entire instrument system used. A GC with dual electron capture detectors is a different technology than a GC with a photo ionization detector or a flame ionization detector. In each case, an analyst would demonstrate proficiency with the specific detector technology employed.

#### **CONTINUING DEMONSTRATION OF PROFICIENCY**

Laboratory proficiency is further demonstrated on a continuing basis through (1) ongoing calibration, (2) method blank analysis, (3) MDL studies, (4) IDL studies, (5) precision and accuracy studies, and (6) analysis of PE samples.

Many elements of the continuing demonstration of proficiency will be completed in the course of routine analyses. Additional work to complete these requirements is only necessary for infrequently performed procedures.

#### **PROCEDURES**

The following components constitute a complete initial demonstration of proficiency within the Lockheed Martin ESAT QA/QC program.

Standard Operating Procedure (SOP) - A SOP that provides all requirements and clear instructions for the analytical procedure.

Initial Instrument Calibration - An initial instrument calibration must be performed complying with the criteria provided in the SOP and reference methods.

Method Blank - A method blank analysis must be completed to demonstrate that the procedure is free of laboratory contamination or to define the background contamination level present in the system.

Precision and Accuracy Study - A precision and accuracy study using four mid-level standards, which are processed through all the sample preparation and analysis steps. In the absence of recommended method acceptance criteria, the laboratory should use recoveries of 70 - 130% as guidance in evaluating the results. One or more analysts may demonstrate proficiency on an instrument through the analysis of the same set of four extracts or digestates for procedures that require a separate sample preparation step prior to instrument analysis.

Method Detection Limit (MDL) - A MDL study, entailing preparation and analysis of at least seven replicates of a low level standard, must be completed. The concentration of analytes in the standards must be approximately three to five times the anticipated MDL. A summary of the results is presented including the analyte name, spike concentration, seven analytical results, average percent recovery, standard deviation of the results, MDL (3.14



times the standard deviation for seven replicates). The calculated MDL must be less than  $\frac{1}{2}$  the method-specified quantitation limit (QL) for each analyte. One set of MDL extracts or digestates may be used on multiple instruments or by multiple analysts to fulfill MDL requirements.

Instrument Detection Limit (IDL) - An IDL study, entailing preparation and analysis of seven replicates of a low level standard (3-5 times the manufacturer's suggested IDL), run three times on non-consecutive days. The result summary includes the analyte name, spike concentration, seven analytical results, standard deviation of the results, IDL (average of the standard deviation for seven replicates). One set of IDL digestates may be used on multiple instruments or by multiple analysts to fulfill IDL requirements.

Performance Evaluation (PE) Sample - A PE sample including at a minimum 10% of the target analytes must be obtained through the appropriate QA personnel, who will evaluate the results based on the predetermined acceptable performance.

Linear Dynamic Range Study - Several inorganic analyses require a linear dynamic range study for each wavelength. This can be performed as part of the initial and continuing demonstration of proficiency. To perform the study, the instrumentation is calibrated as required by the SOP. Next, three to five additional standards, which span the calibration range, are analyzed to demonstrate linearity throughout the dynamic range. Analysts must follow the appropriate reference methods for the QC acceptance criteria as they are method dependent.

#### **DATA INTEGRITY AND LABORATORY ETHICS**

Our program also includes ethics training and the importance of data integrity. The following areas are covered in the analyst initial and continuing demonstration of method proficiency:

1. The use and importance of accurate and complete documentation procedures is explained to each analyst individually and through an annual training seminar.
2. All areas of manual integration techniques and allowable circumstances are discussed through the use of real sample data. Our program also involves specific documentation procedures whenever manual integration is employed. These include before and after printouts, supervisor's signature approval, and notification of the QA department through a written form.
3. All sample data are subject to peer review procedures that include review of the extraction or digestion procedures, standard preparation and calibration evaluation, raw data validation, and calculation verification.

#### **DOCUMENTATION AND RECORD KEEPING**

The laboratory documents all the above activities and the appropriate QA personnel maintain a schedule to assure that each requirement is fulfilled as required. The results of the studies are presented as a summary of target analytes, spike levels, analytical results, percent recoveries, standard deviation of the results, instrumentation description, and analyst information.

Each of the following items is required for both the initial and continuing demonstration of proficiency and must be compiled to document the event:

1. A document signature form that records the analyst has read, understands, and will follow the most current SOP.
2. Initial and continuing calibration information including preparation records, instrument raw data, and laboratory run logs.
3. Method blank data including instrument printouts.
4. Method Detection Limit results in spreadsheet form along with preparation records, instrument raw data, and laboratory run logs.
5. Initial Demonstration results in spreadsheet form along with preparation records, instrument raw data, and laboratory run logs.
6. Performance Evaluation sample results including raw data and true values.

A spreadsheet is used to calculate averages, standard deviations, percent recoveries, MDLs, IDLs, etc. in a standardized format. The QA personnel will have templates available for result reporting.

This information is kept on file and made available for internal and external review.

## SUMMARY

This program has widespread benefits that go beyond our group. With our program, our clients have confidence that the results they are getting are accurate, method compliant, and generated by personnel who have demonstrated proficiency in their respective field. This enables them to go forward with the project or make a decision knowing that they have the best information available.

We believe that the analyst is the main component in the analytical testing procedure. While instrumentation and technology advance, they are only tools which enable a skilled chemist to produce quality work. We trust that this work, produced by proficient analysts, will stand up to the review and examination that today's analytical profession is exposed to.

Lockheed Martin Environmental Services, as a contractor for EPA Region 6, provides support and assistance to the EPA. Our services include laboratory analytical support, data validation, logistics and administration, and air quality monitoring field work under the Superfund program through the Environmental Services Assistance Team (ESAT) contract.

## ACKNOWLEDGMENTS

We would like to thank Tom Chiang and Mike Tindle of Lockheed Martin, ESAT, for their suggestions, recommendations, and peer review.

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## QA DEVELOPMENT FOR PBMS ORIENTED ANALYTICAL OPERATION

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While affecting all the aspects of laboratory operation, PBMS will considerably transform the Quality Systems (QS), as well. Past PBMS implementation experience for commercial operation allows me to evaluate all the complexity of the prescriptive methods - PBM transition process.

Current Quality Systems (QS) are designed primarily for the commercial environmental laboratories with the mandated methods based operations. After establishing successful NELAC compliant QS for the commercial analytical laboratory in Louisiana, I've experienced substantial resistance trying to blindly copy it for university based (UB) center. Analysis of the roots of this resistance enables me to identify the following unique features of the academia based facilities:

1. UB analytical operations focus their infrastructure on PBMS, rather than prescriptive methods application;
2. UB projects may not incur significant legal liabilities.
3. Academia culture organically incorporates PBM needs for non-routine, creative approach;
4. In order to meet specific research/project requirements UB environmental centers regularly foster new technology development and analytical methodologies modification and improvement, generating technically defensible analytical data, as well.
5. Laboratory capabilities may be expanded due to cooperation with other university departments and facilities;
6. PBM implementation will change the projects' DQO process emphasizing the need to plan the entire sampling, measurement and assessment process up front before any field activities begin (6). As a rule, the university laboratories plan and conduct all phases of the projects, from it's design to results' interpretation.

Following, several PBM development and implementation schemes in the UB setting will be presented as case studies. QS changes necessary to support PBMS process will be discussed.

Reiterating all the factors mentioned, the academia based environmental analytical facilities at present time possess the necessary infrastructure and are ideally positioned as the models for PBMS principles development and application.

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### **USE OF RECORDABLE MEDIA FOR ENVIRONMENTAL ELECTRONIC DATA VALIDATION IN THE PAPERLESS LABORATORY**

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The Lockheed Martin Environmental Services Assistance Team (ESAT) at the Region 2 United States Environmental Protection Agency (EPA) assists in the analysis of samples collected from Superfund and Brownfield sites. ESAT provides legally defensible data analyses used for human and environmental assessments. The reporting of the analyses take the final form of "data deliverables" which are cumbersome and difficult to review in their current paper format. In order to facilitate the review and reporting function, a comprehensive system is needed to replace the paper trail with an electronic equivalent. This system would streamline the process without compromising data validity, increase productivity, and minimize issues of error and fraud.

Currently, no definitive standard exists for the creation of such a system although concepts under the Good Laboratory Practices (GLP) program have addressed data validation issues. Computerized systems used to capture, reprocess, report or store raw data electronically must provide for the retention of full audit trails. Any reprocessed data must be traceable to the person(s) by use of timed and dated (electronic) signatures without obscuring the original data. Reasons why any data was reprocessed, e.g., instrument variations, should also be stated.

A method was developed to aid in the reviewing and reporting of the large amounts of data associated with even the smallest of projects. This is made feasible with refined recordable compact disc technology (CD-R) and data processing capabilities at remote computer stations. The data is collected and stored initially on the instrument computer. Upon cursory analyst review, the data and all quality assurance data is transferred to a CD. The data reviewer thus receives a data package consisting of a summary of results, QA/QC summary and index of the raw and supporting data contained on the CD. An autonomous data review software package can simulate the data work up remotely at the data reviewers site enabling efficient and simple review of proper manual integrations, QA criteria, and qualitative and quantitation results.

Once reviewed and validated, the data is transferred for network archival either directly into a Laboratory Information Management System (LIMS) or any other data storage system. In addition, the completed "data deliverable" may be submitted electronically to the client creating a paperless laboratory scheme.

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### **DUPLICATES AND/OR SPLIT SAMPLES ARE OFTEN TAKEN IN ENVIRONMENTAL PROGRAMS**

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Duplicates and/or split samples are often taken in environmental monitoring programs and sampling studies. The intended purpose of these data is usually as a check on laboratory performance during a week or other sub period of the entire measuring period. However, it seems that if such samples are actually random samples of an entire monitoring or study period or entire study that such data would provide a valid means of making inferences to the study population concerning the measurement error that existed during the whole period. This information would be useful in the subsequent analysis of the data and for comparisons between studies. An example of a method to